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DEVELOPMENT OF INSTRUMENTATION FOR QUANTITATIVE COLLECTION OF TOTAL ATMOSPHERIC MERCURY FROM AMBIENT AIR

D. J. Sibbett, et al

Geomet, Incorporated

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A prototype device for the quantitative collection of airborne mercury in particulate, elemental and combined forms, respectively, was designed, fabricated and tested. The device utilized a Hi-Vol Sampler into which two canisters containing absorbers for elemental and combined mercury were added in a collection plenum below the glass fiber filter. Particulate collection was tested with mercuric oxide and sulfide on the glass fiber filter; elemental mercury vapor was collected on a silver-alumina absorbent; dimethyl mercury was collected on the activated charcoal phase. The absorbents and canister configuration are described. The absorbents were performance tested in 24-hour runs at challenge levels from ambient to 118 micrograms/m^3. The collection efficiency of both silver on alumina and activated charcoal media exceeded 99.9+ percent. An analytical procedure for each of the three separately collected forms of mercury has been developed and tested. In principle, it involved desorbing the collected sample into iodine monochloride solution by application of heat to the sample; reduction of an aliquot of the ICl solution; collection of the resultant elemental mercury on gold wire; heating the gold wire by an induction furnance to desorb the concentrated mercury into a flameless atomic absorption spectrophotometer cell four measurement.

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- Spectrophotometers
- Mercury
- Design
- Charcoal
- Absorbents
- Filter materials
- Plenum chambers
- Iodine halides
- Vapors
- Tests
- Standards

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- Silver treated alumina pellets
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24 June through 23 December 1972

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Section 1.0

INTRODUCTION

GEOMET, Incorporated submits the draft of the Final Report under Contract 68-02-0578 in accordance with the Reports of Work Requirements as stipulated by the contract. This report provides a description of the effort expended over the period of 24 June through 23 December 1972.

The objective of the program for Development of Instrumentation for Quantitative Collection of Total Atmospheric Mercury from Ambient Air was to design, develop and fabricate a prototype collection device for the quantitation of mercury in air in the three major forms: (1) inorganic and organometallic particulates, (2) inorganic and organometallic vapors, and (3) elemental mercury at levels up to a threshold limit value of 100 micrograms/M^3. The collection device(s) and attendant processing techniques are compatible for utilization in or with Hi-Vol samplers of the National Air Survey Network for air sampling in the field. Design of the collection devices is oriented to obtain quantitative mercury retention, to give convenient packaging, storage and shipping, and to give ease of analysis at centrally located laboratories after use.

The scope of the contract effort included the design, fabrication and testing of prototype collection devices, adaptation of those devices to Hi-Vol samplers, provision of techniques and processing equipment for analysis of the collected mercury forms, full system quantitative testing, and delivery to EPA of the set of prototype instrumentation following a demonstration of the effectiveness of the system. The overall aim of the program was to provide qualified prototype instrumentation capable of being put into the field on an economical large-number basis.

Section 2.0

PROGRAM ACCOMPLISHMENTS

The effort expended under this program covered the development of a Prototype form of the mercury collection device. The collection method allows the total and quantitative collection and separation of the three forms of mercury - (1) particulate, (2) elemental and (3) inorganic and organomercury vapors. These individual samples may be easily packaged and transported back to some centralized laboratory for analysis. The analysis measures the mercury content in each collected solid phase, corresponding to the forms indicated above and thus provides total mercury values regardless of the mercurial forms present in the sampled ambient air.

2.1 TECHNICAL APPROACH

The technical approach for the Development of Instrumentation for Quantitative Collection of Atmospheric Mercury from Ambient Air was designed to meet all the requirements stipulated by the EPA in Contract No. 68-02-0578. The program was carefully structured to be compatible with the phases contained in the Contract Scope of Work (Reference Section 2.2 below). In addition, the program was oriented to provide the necessary technical data from practical testing of hardware developed under the contract.

Specific areas of development effort were 1) the minimal modifications to a standard Hi-Volume air sampler while incorporating the new instrumentation, 2) definition of collection canister configuration and selection of absorbents for all forms of mercury, and 3) utilization, as a baseline configuration, of existing chemical and electromechanical devices for analysis of collected mercury samples. A stepwise approach to the solution of these problems was created in order to optimize the accomplishments during this short duration program.

Hi-Volume Sampler Modifications

In accordance with the contract Scope of Work, preliminary design concepts relating to the collection system evolved around the insertion of collection canisters into the body of a typical Hi-Vol sampler. This concept precluded any physical change to the sampler itself, but did increase the sampler height. It is expected that this added canister plenum, incorporating threaded ring fittings, will be adaptable to all commercially available Hi-Vol samplers being marketed in the continental United States. The only remaining assembly criteria is the varied construction details of shelters in which the Hi-Vol samplers are operated. GEOMET chose a UNICO 550 Turbine-Jet High-Volume Air Sampler (marketed by the Environmental Science Division, Bendix Corporation) for use on this program. This particular sampler is housed in a rather inexpensive plywood shelter. Since the sampler is supported in place by the upper edge of the incoming air funnel duct, the Hi-Vol and collection plenum now extends below the sides of the shelter. This in no way affects the operation of the sampler, but does allow the Hi-Vol air through-put gage to be somewhat exposed to the open air and brings the exhaust port closer to the ground. GEOMET also owns a High-Volume sampler marketed by the PRECISION Scientific Company. This Hi-Vol is supported by a ring around the blower motor housing, rather than by the air inlet ducting. When installed in the supplied aluminum shelter, the Hi-Vol and plenum system protrudes above the shelter at the top prohibiting positioning of the hinged roof-cover. It is, therefore, concluded that while the simple utilization of the collection plenum obviates any modification to the Hi-Vol sampler, some slight modifications may have to be made on specific Hi-Vol sampler shelters currently in use in the National Air Sampling Network (NASN). Any modifications would consist of increased side protection and be dependent on the type of shelters in use.

Details of the collection sample plenum for the Hi-Vol samples are included in Section 2.3, below.

Collection Canister Configuration and Absorbents

In the proposal for this program, GEOMET submitted conceptual designs for the Prototype instrumentation. As a part of the total system, the idea of using stacked "canisters" for positioning and support of the mercury absorbents with the Hi-Vol was established. During the program, various developmental parameters were exploited and the Prototype design evolved as a result of testing several configurations. Several limiting factors became readily apparent. The maximum height and diameter of two or more collection canisters was determined by Hi-Vol sample air through-put, necessary adsorbent volume as determined by the collection efficiency of the test absorbents, and the wide range over which quantitative mercury collection must be made. The final configuration of the canisters was predicated on results of testing to explore these limiting factors. The rationale for canister size, while tied directly and unequivocally to absorbent bed size, was to provide space for enough absorbent (in the elemental mercury collection canister) to cover mercury levels spanning five (5) decades, i.e. from 1 ng/M^3 to 100,000 μg/M^3 of mercury. Also, the constraint of final analysis of the sample by commercially available instruments was superimposed onto the canister design calculations, and subsequent testing was utilized to ascertain the minimum canister sizes which would provide enough sample of any range of collected mercury for meaningful analysis. Of lesser importance, was the actual canister configuration. Here items such as materials of construction, handling and packaging were investigated. The collection canister design for the Prototype system is a result of the investigation of these factors. Further details of the current design are found in Section 2.6.3 and 2.6.4 of this report.

Absorbent development was based on the premise that both EPA and GEOMET wanted several non-sole source commercial suppliers of absorbent for the Prototype system. GEOMET examined several potential sources of commercial collection media and purchased batch lots of treated aluminas and charcoals for the collection of elemental and organic mercury. Finally, GEOMET set out to manufacture batch lots of treated alumina for elemental mercury collection. None of the tested, commercially available absorbents were comparable in performance to the GEOMET product. Therefore, all elemental mercury collection made during the final test periods of the program were made with the GEOMET prepared absorbent. Developmental details of this effort are found below in subsections of 2.6.

Analysis of Collected Mercury Forms

GEOMET utilized, as a baseline system, the wet chemistry method for determination of mercury as described in the Federal Register, Volume 36, No. 234 - Tuesday, December 7, 1971. GEOMET did, however, institute unique pre-processing steps as well as add other types of instrumentation between the liquid impinger system and the Atomic Absorption Spectrophotometer (AAS). A special technique was also developed for the analysis of particulate mercury found on the Hi-Vol filter. The analysis procedures finally developed, while still in the prototype stage, fully satisfied the needs of the program as specified in the Scope of Work and appear to offer an accurate and precise method of rapid testing of samples. Specific procedural steps are found in Section 2.7 below.

2.2 PHASED FULFILLMENT OF PROGRAM GOALS

In Exhibit "A" of the contract, Scope of Work, the Environmental Protection Agency provided a program effort outline divided into five (5) Phases. Inasmuch as this phased effort has considerable overlap in tasks between phases, GEOMET reordered some of the tasks for a more chronological approach to the development of the instrumentation. In order to clearly demonstrate the GEOMET response to the Scope of Work, the following table is provided which indicates Phase task and the corresponding GEOMET development. Reference is also provided to Sections of this report wherein additional data may be found.

Table 2-1 Phase Program Goals

Scope of Work:	
Phase I	**GEOMET Response**
The Contractor shall design, fabricate, and evaluate a prototype device for the quantitative collection of airborne mercury and its compounds (inorganic salts and organometallic vapors) from the ambient air. The device shall have the following performance characteristics:	
A. The prototype device shall be constructed for ease of handling in field operations and shall be simple and compact in structure to facilitate low cost shipment by mail to central laboratories following sample collection.	The collection canister design is simple, inexpensive and compact. The plastic cylinders may be reloaded in the field or mailed to laboratories in commercially available containers. (Section 2.6, 2.7, and 3.0)
B. It is desired that the collection device be used with the Hi-Vol samplers of the National Air Survey Network as an attachment to their present collection system.	The collection plenum inserts into the Hi-Vol sampler without modifications to the Hi-Vol or use of special tools. (Section 2.7.2)
C. Construction materials shall be economical, to allow for eventual production of large numbers of the collectors for nationwide surveys.	Initial collection plenum construction is of aluminum. Prototype canisters are of PVC plastic. It is conceivable that ultimately all parts could be fabricated from low cost plastic materials. (Sections 2.7.2 and 3.3)
D. The materials of construction shall have a low mercury background and be readily cleaned of any mercury collected.	Current materials are free from mercury, and it is not apparent that these materials are absorbing mercury during sample testing. (Section 2.7)

Table 2-1 Phase Program Goals (Con't.)

	Phase I (Con't.)	GEOMET Response (Con't.)
E.	The collection device shall be capable of retaining the collected mercury in all its forms in a stable state for several weeks and to quantitatively release the mercury for analysis.	Absorbents clearly absorb the levels of mercury stipulated by the Contract. Collection efficiency runs show that some slight difficulty remains in uniform recovery of all absorbed mercury vapor. Careful packaging will hold samples in stable state for several weeks. (Section 2.8.3)
F.	The collection device shall be capable of quantitatively collecting all forms of mercury from the ambient air at flow rates common for the Hi-Vol sampler (20 CFM or better). Its capacity for retention of mercury and its compounds should be for all mercury present in a 24-hour consecutive sampling period. This requires the capability of handling mercury concentration ranges from the natural background levels of a few nanograms per cubic meter to the threshold limit value of 100 micrograms per cubic meter.	The collector separates particulate, elemental and combined mercury (vapors) onto a glass fiber filter, a silver/alumina adsorbent and activated charcoal, respectively. Sampling rates of 20 CFM or better have been utilized. Several long duration sample runs at Hi-Vol through-put of ≥ 20 CFM were made during the course of the program. Challenges varied from 4.5×10^{-2} $\mu g/M^3$ (elemental) to 118 $\mu g/M^3$ (organic). These combined mercury runs are reported in Section 2.9.
G.	The collection device may consist of several stages or compartments with each specific for a particular form of mercury found in the ambient air. The three forms of mercury to be collected separately are elemental mercury, inorganic and organometallic mercury in particulate form, and inorganic and organometallic mercury vapors.	The Prototype system utilizes a standard Hi-Vol fiberglass filter for inorganic and organometallic particulate forms, a canister stage for elemental mercury and for inorganic and organometallic vapors. (Sections 2.6 and 2.7)

Table 2-1 Phase Program Goals (Con't.)

Phase I (Con't.)	GEOMET Response (Con't.)
H. The collection device shall be able to separate for independent analysis the three major forms of mercury (elemental, particulate, organic vapors).	Collection stages (previous page) hold each form of mercury in a separate state for independent analysis. (Sections 2.6, 2.7 and 2.9)
I. A removal procedure for the collected mercury shall be developed so that the mercury can be analyzed by readily available analytical instruments, both inexpensive and sophisticated.	GEOMET has developed unique apparatus and/or methods for recovery analysis of collected mercury. See Section 2.9.
J. A delineation of the types of instruments that can be used for these analyses and proof of their capabilities shall be prepared and submitted to the Project Officer.	Instruments, by type, for similar processing are noted in Section 2.8.6.
Phase II	
The fabricated collection device shall be checked to establish its efficiency for collection of airborne mercury and its compounds. Known mercury standards in several chemical forms (elemental, inorganic, organometallic) shall be used to determine their collection characteristics. Modifications should be made to optimize the collection efficiency of each species. Several environmental factors shall be checked for their influence on the collection efficiency of the device. These are climate factors such as temperature in the temperate range, moisture, dust load, and winds. Other factors like gaseous pollutants (H_2S, SO_2, NO_x, phenols hydrocarbons) shall be checked for their effect on the collection efficiency for mercury.	The Prototype system was effectively challenged by varied amounts of elemental vapors, organic mercury, and solid particulate mercuric compounds. Collection efficiency runs were carried out and reported on a monthly program basis. Contaminant gases were superimposed over the combined mercury form challenges. Limited ambient air challenges were carried out with the total system. (Section 2.9-all subsections.)

Table 2-1 Phase Program Goals (Con't.)

	GEOMET Response
Phase III	
A transfer step to remove the collected mercury from the collector to an appropriate analytical instrument is envisioned. The step could require the development of a handling system for transfer of the mercury vapor. If such a system is developed, it shall be tested for quantitative transfer and clean-up to the previous background levels for mercury.	GEOMET utilized a modified Hatch and Ott procedure for processing the elemental and organic samples through the analysis procedure. Special handling and processing components were fabricated to aid in the analysis of the adsorbents. (Section 2.8).
Phase IV	
Detailed scientific data shall be furnished to support all claims on the efficiency of mercury collection. Similar data shall document the effect of atmospheric variables and gaseous pollutants on the collection efficiency. Prototypes of the developed collection device and any transfer system shall be furnished.	Comprehensive testing of the Prototype system was carried out, including several long duration (≥24 hours) runs. The results of these tests are discussed and tabularized in the subsections under Section 2.9.
A tabulation of the specifications of all equipment developed under this contract shall be submitted to the Project Officer with the final report on this project. If a transfer or sample processing step is necessary before the mercury sample can be analyzed, a written procedure for this operation shall be furnished with the final report.	The tabulated equipment list is presented in Section 4.0. Procedures for sample analysis are discussed in Section 2.7.2.

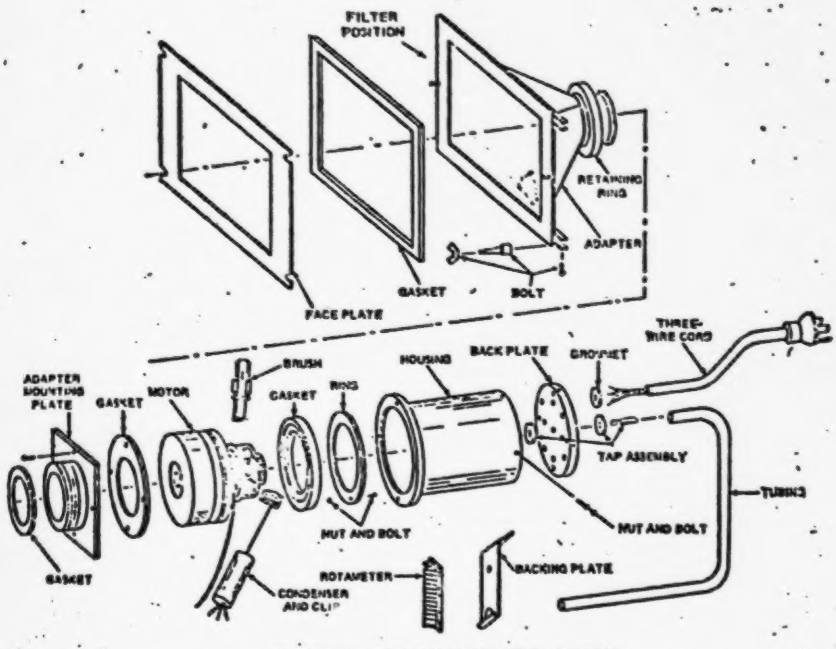
Table 2-1 Phase Program Goals (Con't.)

Phase V	GEOMET Response
At the completion of the effort prescribed hereinabove, the Contractor shall conduct a demonstration of the operation of the collection device developed under this contract before the cognizant Project Officer at the National Environmental Research Center, Research Triangle Park, North Carolina 27711.	GEOMET, with permission of EPA, demonstrated the Prototype system and procedures to the Program Monitor at the GEOMET laboratory in Pomona, California. (Section 2.7.5)

2.3 HIGH-VOLUME AIR SAMPLER SELECTION

GEOMET purchased and has operated a Unico 550 Turbine-Jet High-Volume Air Sampler (Environmental Science Division, Bendix Corporation) throughout this program. The Unico was chosen due to the simplicity of design of the sampler and of the sampler shelter. The sampler is hung in the wooden shelter and supported by the lower edge of the inlet ducting adaptor which contains the particulate filter. A typical sampler of this configuration is shown in Figure 2-1. The originally proposed concept was to place a collection plenum between the incoming air duct, and the sampler blower motor casing. The Unico 550 lends itself quite suitably to this arrangement. The plenum elongated the blower/ducting dimension, but does not affect the sampler operation nor cause any modification to the sampler shelter. The addition of the plenum to the Unico 550 is shown, in a conceptual sketch, in Figure 2-2A.

GEOMET also owns a Precision Scientific Hi-Vol sampler, which is offered with an optional aluminum shelter. While the shelter for the Precision sampler is well built and durable, the sampler/shelter combination requires modification to add the GEOMET collection plenum. This sampler is held in place by a notched shelf built into the shelter. The Precision Hi-Vol contains a cast ring which is an integral part of the sampler blower motor housing. The ring rests on the shelf and supports the sampler within the shelter (Figure 2-2B). With this particular unit, the collection plenum cannot be inserted into the sampler system without either obviating the use of the shelter roof, or lowering the shelter sampler support shelf to allow clearance for the Prototype system.


FILTER POSITION
RETAINING RING
ADAPTER
GASKET
BOLT
FACE PLATE
THREE-WIRE CORD
ADAPTER MOUNTING PLATE
GASKET
MOTOR
BRUSH
GASKET
RING
HOUSING
BACK PLATE
GROMMET
TAP ASSEMBLY
TUBING
NUT AND BOLT
NUT AND BOLT
GASKET
ROTAMETER
BACKING PLATE
CONDENSER AND CLIP

Exploded view of typical high-volume air sampler parts.

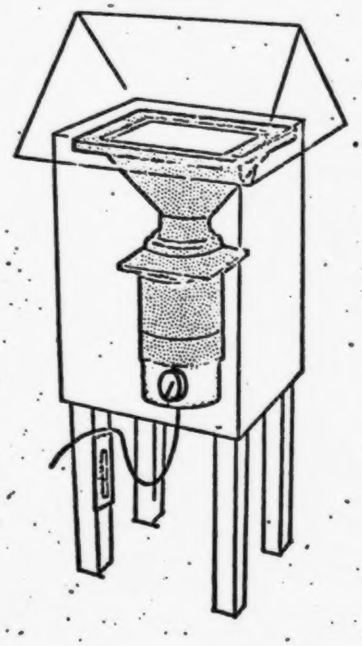
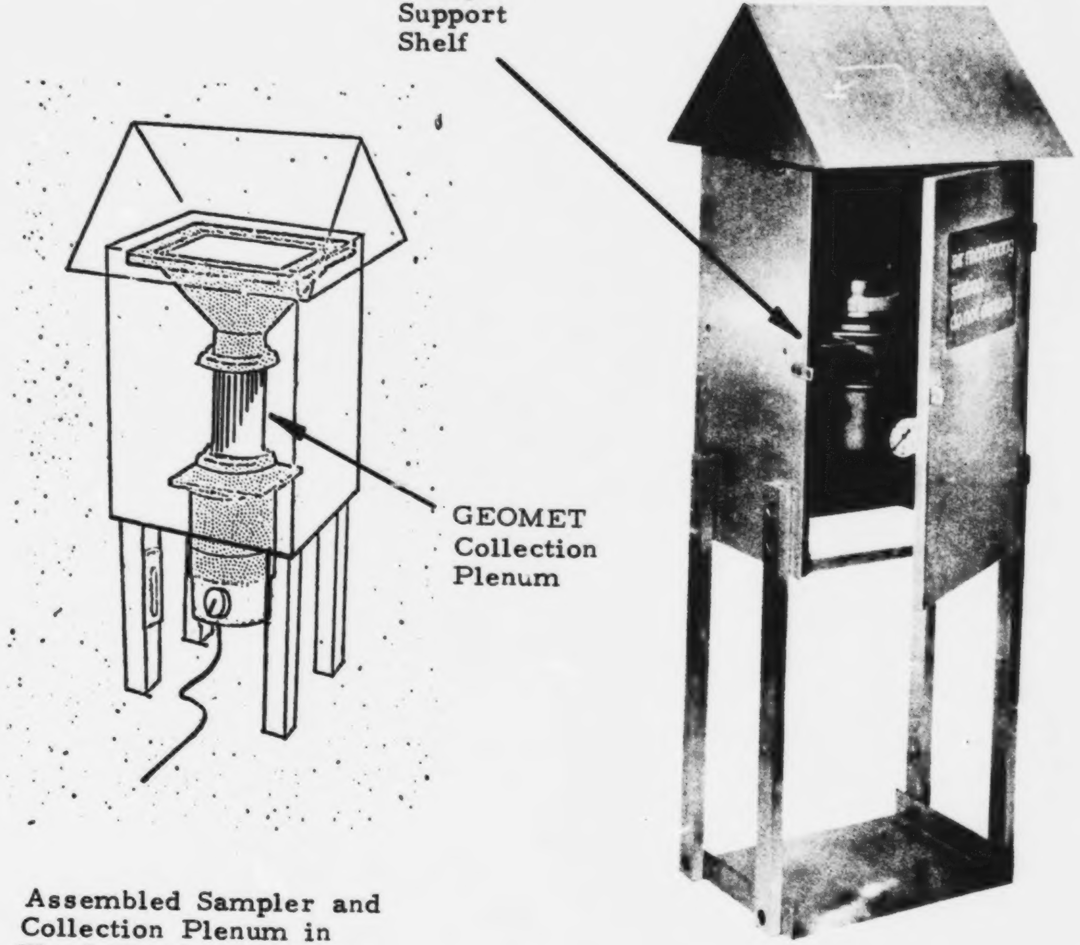


Figure 2-1. Assembled Sampler and Shelter.


Sampler
Support
Shelf
GEOMET
Collection
Plenum

A. Assembled Sampler and Collection Plenum in Wood Weather Shelter

B. Precision Scientific High-Volume Sampler and Aluminum Shelter

Figure 2-2
Commercial Hi-Vol Sampler

Two points are evident as a result of this discussion: 1) the Unico 550 sampler and shelter were well chosen for this program, and 2) thought should be given to the varied configurations of sampler shelters already in the field. It is assumed that some standard will be set on High-Volume sampler configuration, or that the simple modifications necessary to accept this Prototype instrumentation will be allowed.

2.3.1 Hi-Vol Sampler Air Through-put Studies

The first work done with the Unico 550 Hi-Vol sampler was to run preliminary studies on air through-put of collection materials and collection canister configurations.

Various mesh sizes of charcoal and alumina were tested in a cylindrical tube mounted in the annular throat of the Hi-Vol filter adapter ducting. Barnebey-Cheney 6-10 mesh activated charcoal was tested, as well as the following alumina material - Harshaw 0.125 inch diameter pellets, Alcoa 4-8 mesh and Alcoa 8-14 mesh granules. It was determined that up to 3.75 inch bed depths of these materials could be loaded in a 3.0 inch average diameter tubular canister, and a nominal value of 20 CFM could be easily pulled through the Hi-Vol sampler. Later, up to seven inches of alumina (0.125" pellets) were utilized with the Hi-Vol, including a fiberglass particulate filter, and a nominal value of 20 CFM through the Hi-Vol was maintained.

Through-put studies were also made of a prototype canister made of perforated stainless steel. A 400 gram batch of alumina pellets (~3.50 inches deep in a 3.1 inch diameter canister) which had been tested in the Hi-Vol at 20 CFM, gave 35 CFM through-put when transferred to the prototype perforated canister. These canisters, containing eight 0.062 holes per linear inch (64 per inch^2) were considered for final canister configurations wherein collection media density or shape required large volumes of the collection material and low residence time of the through-put sample air stream. These early Hi-Vol tests outlined the magnitude of the amounts of adsorbents which could be used to collect mercurial vapors. The ultimate sizing of the canister beds is discussed in Sections 2.6, 3.3 and 4.0.

2.4 MERCURY CHALLENGE SOURCES

In order to evaluate the interim design concepts and components prior to the design of the Prototype system, methods of providing accurate amounts of the three forms of mercury were necessary. These methods and/or apparatus required capability of supplying mercury challenges to the collection system with known mercury concentrations and minimum loss. The minimization of loss was a very important aspect of the total collection efficiency of the Prototype system. A review of the challenge methods is presented in the following sections.

2.4.1 Particulate Mercury Challenge Sources

GEOMET utilized ultra-fine mesh mercuric oxide (HgO) and mercuric sulfide (HgS) to challenge the collection efficiency and particle retention of the standard fiberglass Hi-Vol Sampler filter. All runs on this program were made using the Reeve Angel 934-A12 (8" x 10") standard Hi-Vol filters. It was discovered early in the program that in order to provide even distribution of the challenge material over the filter area, the mercuric compounds must be mixed with some inert carrier. This was especially true at low challenge levels wherein less than 1.0 μgm of material was to be used. To prevent as much loss of the challenge sample as possible, the weighed aliquots of HgO or HgS were placed into $\leq$5 gram batches of Cab-O-Sil, an inert fumed silica. This mixture was then fed into the Hi-Vol sampler air stream and onto the filter. This method was extremely successful for the application of particulate challenges to the Prototype instrumentation system.

2.4.2 Elemental Mercury Vapor Source

Four separate source models were constructed in the attempts to assemble a device for challenging prototype collection canisters with elemental mercury. Early attempts were made to devise a method for determination of mercury vapor emissions through weight loss measurements. Considerations of the very small amounts of mercury to be measured caused the abandonment of this procedure. The ultimate method devised, which has been completely successful, utilized air passage over heated liquid mercury under controlled conditions. This apparatus is depicted in Figure 2-3.

In operation, air is pumped through the sealed vapor source system and passes over a small pool of liquid mercury. Heating tape is used to heat the mercury held in the U-tube increasing the mercury vapor pressure for high challenge levels. Voltages

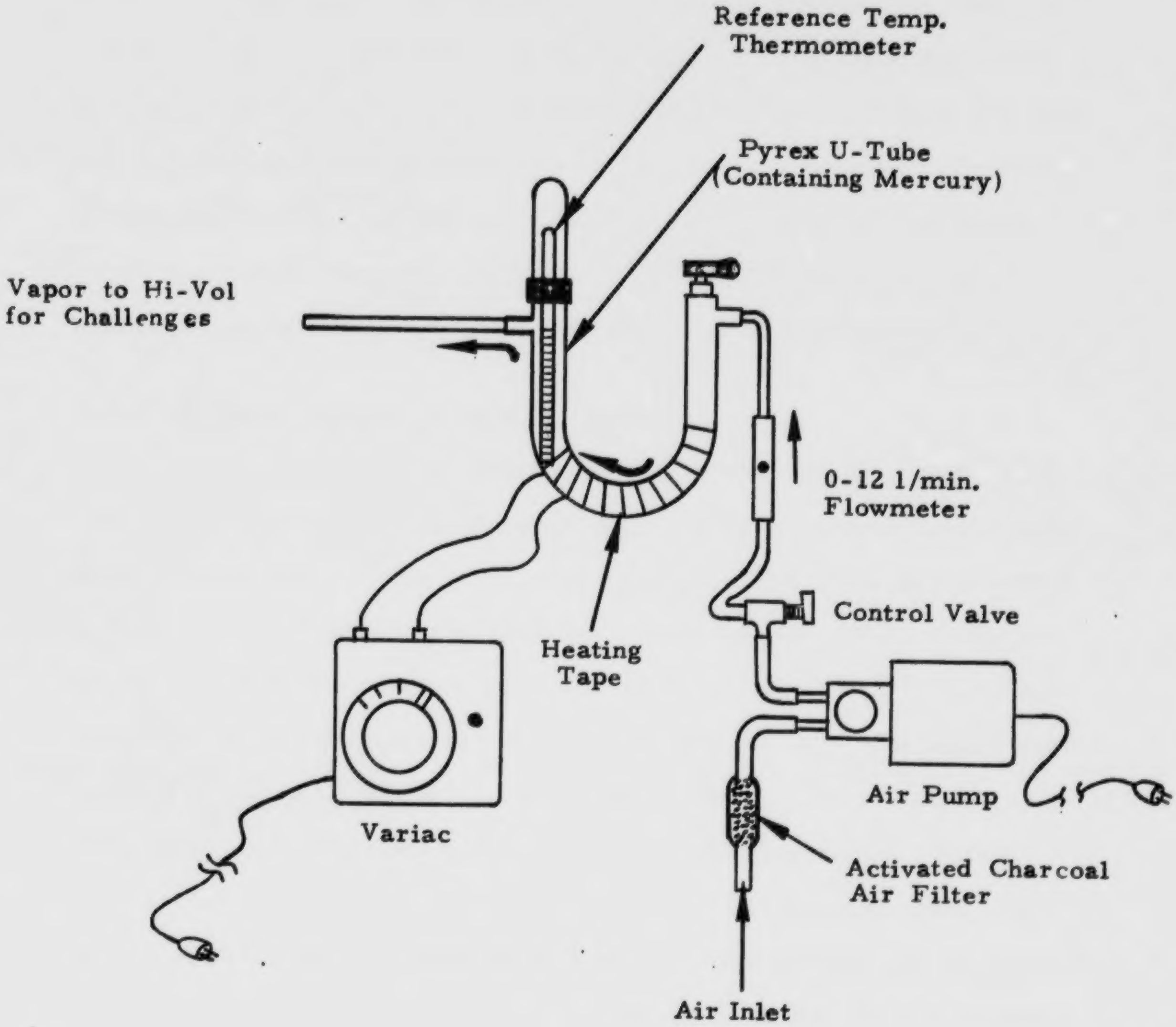

Reference Temp. Thermometer
Pyrex U-Tube (Containing Mercury)
Vapor to Hi-Vol for Challenges
0-12 l/min. Flowmeter
Control Valve
Heating Tape
Variac
Air Pump
Activated Charcoal Air Filter
Air Inlet

Figure 2-3 Elemental Mercury Vapor Source

from 0-60 VAC yield challenges of from 0.3 to >200 μg/M^3 (at temperatures ranging from ~20°C to ~120°C). All data was taken with air flow through the U-tube at 5 liters/minute, but both air flow and temperature can be varied for a wide range of values. Testing proved the success of this method in providing elemental mercury vapor challenges over the complete spectrum of mercury levels required for quantitative testing under the scope of this program.

2.4.3 Organic Mercury Challenge Sources

Several challenge methods were evaluated for organic mercury input sources during this program. Early attempts using commercial mercury sources were unsuccessful. For example, a diethyl mercury Dynacal permeation tube was acquired from Metronics Associates. It was Certified to permeate at a rate of 264 ±5% ng/minute at 40°C. This special-order device was utilized in a Metronics heat exchanger and tube holder, as depicted in Figure 2-4. Challenge runs were attempted utilizing a 200 gm. collection canister filled with sulfur treated (13%) activated charcoal. Permeation tube challenges at 40°C were monitored with a GEOMET Module 109 Catalytic Converter in conjunction with the Model 103 M.A.M. It was rapidly apparent that the permeation tube was releasing elemental mercury vapor into the tests rather than diethyl mercury, exclusively. Apparently, the diethyl mercury in the permeation tube was partially hydrolyzed or decomposed. For the purpose of testing adsorption on the sulfur treated charcoal, this mixed source was not satisfactory. No further testing was attempted with this prototype permeation tube.

Figure 2-4

METRONICS®

Technical Bulletin No. 7-70

DYNACAL® PERMEATION TUBES

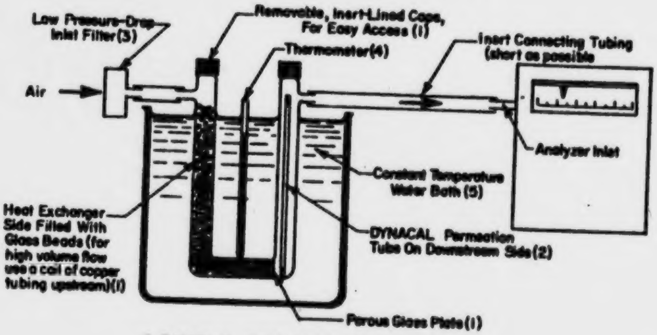
APPLICATION NOTE ON THE TESTING AND CALIBRATING OF AIR ANALYZERS

Testing

An air analyzer can be quickly tested to see if it is "about right" by placing a DYNACAL® permeation tube into the analyzer's inlet air stream. One satisfactory test method is to put the DYNACAL® tube into a cylinder such as a 20-cm glass drying tube and connect the lower end of the drying tube to the analyzer inlet. Clean and empty gas absorbtion tubes (plastic) work equally well. Temperature is obtained from a thermometer placed near the inlet. Concentration is estimated using the DYNACAL® standard rates for the particular temperature and tube length.

Calibrating

A DYNACAL® permeation tube and the Metronics specially constructed 2-section glass apparatus can be used as shown below to provide a gas stream of known concentration for dynamic calibration.


Low Pressure-Drop Inlet Filter(3)
Removable, Inert-Lined Caps, For Easy Access (1)
Thermometer(4)
Inert Connecting Tubing (short as possible
Air
Analyzer Inlet
Constant Temperature Water Bath (5)
Heat Exchanger Side Filled With Glass Beads (for high volume flow use a coil of copper tubing upstream)(1)
DYNACAL Permeation Tube On Downstream Side(2)
Porous Glass Plate(1)

A SIMPLE CALIBRATION APPARATUS

Parts List

1. Heat exchanger and tube holder with with glass beads available from Metronics. Model 4-15 holds 4 ea. 15-cm tube
   Model 4-30 holds 4 ea. 30-cm tubes
2. DYNACAL® tubes available from Metronics. Please see other side for HOW TO ORDER.
3. Inlet filter similar to GCA Chemical Cartridge and end-of-line holder available from Mine Safety Appliance, 201 N. Braddock Ave., Pittsburgh, PA 15208 is adequate for most purposes. Cylinders of zero-air or nitrogen are recommended where background contamination is significant.
4. Thermometer, high grade laboratory type of suitable range and graduation, is available from most laboratory supply companies.
5. Constant temperature bath can range from tap water bath (about 17°C in our local system) to baths complete with precision temperature controllers such as those sold by Cole-Parmer, 7425 N. Oak Park Ave., Chicago, Ill. 60648.

METRONICS ASSOCIATES, INC. 3201 PORTER DRIVE · STANFORD INDUSTRIAL PARK · PALO ALTO, CALIFORNIA 94304

Diluted diethyl mercury in CCl_4 was also briefly utilized to assess the collection on sulfur-treated charcoal. However, again decomposition or hydrolysis appeared to interfere. Utilizing the Mercury Monitor separately and in conjunction with the Catalytic Converter, it was estimated that the diethyl mercury had decomposed into a complex 47.5% elemental mercury and 52.5% $(C_2H_5)_2Hg$. Reagent grade diphenyl mercury was also examined as a challenge material. However, the vapor pressure of this compound at room temperature is too low to be effectively used. When the sample was heated, it sublimed. Condensation of the sublimed vapors throughout the apparatus made quantitation impossible and left objectionable contaminating residues.

A supply of dimethyl mercury was then obtained, and used as the challenge form for organic mercury in tests throughout the remainder of the program. The dimethyl mercury was held in sealed containers, and diluted in 1-propanol to the required challenge concentration immediately prior to each test run. The 1-propanol was chosen due to its high boiling point (97.2°C). The challenge mixtures were applied to the Hi-Vol by placing the challenge mixture container in the incoming air stream, and evaporating the mixture into the Hi-Vol from a wick-feeder protruding from the container. The liquid challenge volume was preset to be totally evaporated during the test period. If there was a residual challenge volume, it was measured as accurately as possible and subtracted from the anticipated calculated challenge level.

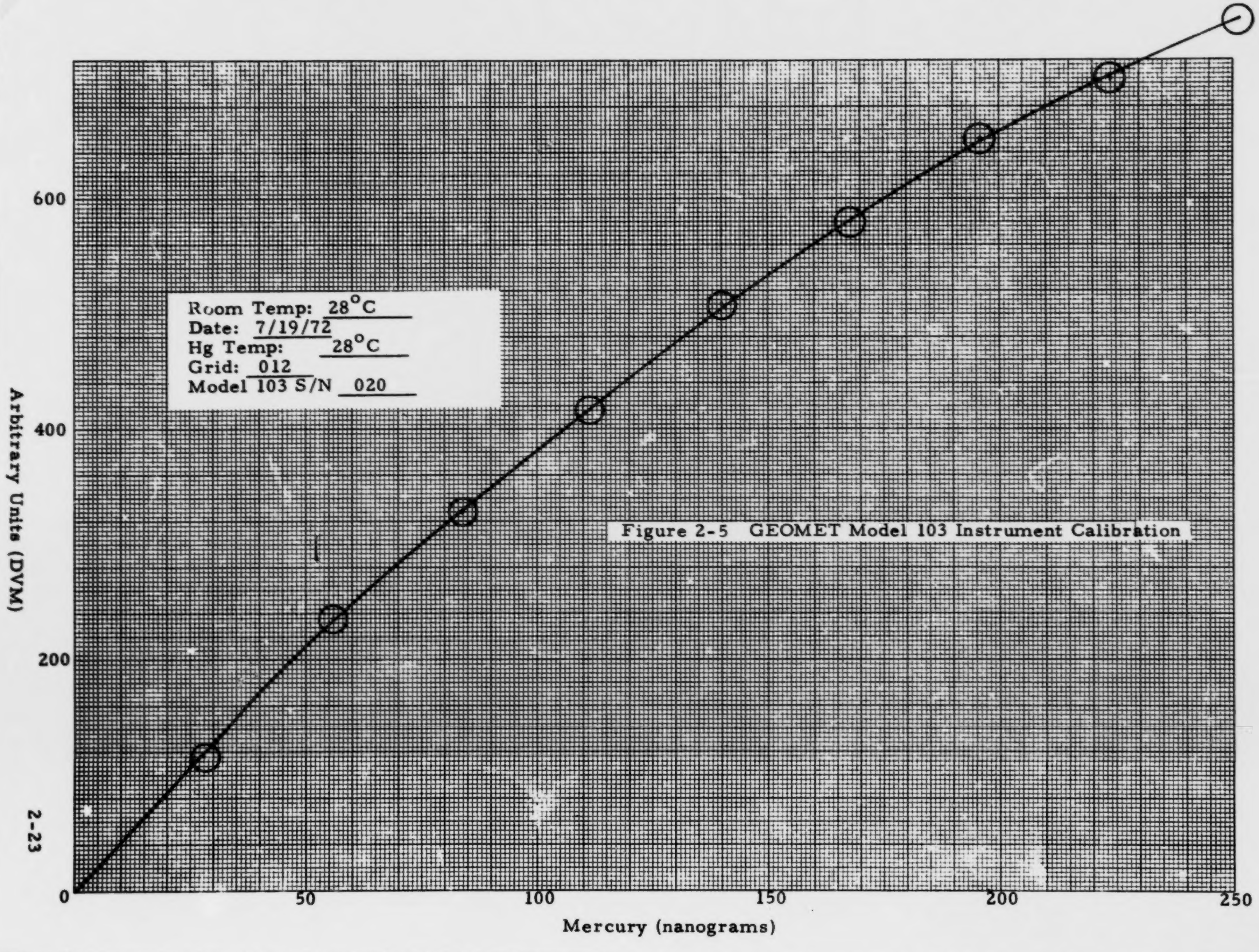
2.4.4 Ambient Air Challenges

Some ambient level of elemental mercury was always present during the test runs carried out during the program. Background adjustments were made to compensate the collected data for this effect.

At the end of the program, a final long duration ambient air run was made utilizing the final Prototype collection system. In this run, no "artificial" mercury challenge was added over the normal mercury level found in the Pomona, California atmosphere. Results of this test are presented in Section 2.9.7.

2.5 AIR TRAIN CHALLENGE APPARATUS

In order to facilitate near real-time measurements of mercury vapor challenge levels, as well as obtain collection efficiency data on collection canister configurations under test, it was decided to utilize the GEOMET Model 103 MAM as an ancillary device within the air train challenge system. The data collected with the M103 was then used, in later recovery analysis runs, to correlate the calculated mercury challenges on collection canister absorbents. This data was correlated directly with an atomic absorption spectrophotometer (AAS) (Perkin-Elmer Model 303). The Model 103 was set up and operated with the mercury vapor source described in Section 2.4.2 above. A part of the checkout procedure was a baseline calibration of the instrument. A typical graph of that calibration is provided in Figure 2-5. A sampling probe was installed to remove approximately 1 CFM of the air passing through the Hi-Vol sampler and supply that



Room Temp: 28°C
Date: 7/19/72
Hg Temp: 28°C
Grid: 012
Model 103 S/N 020

Figure 2-5 GEOMET Model 103 Instrument Calibration

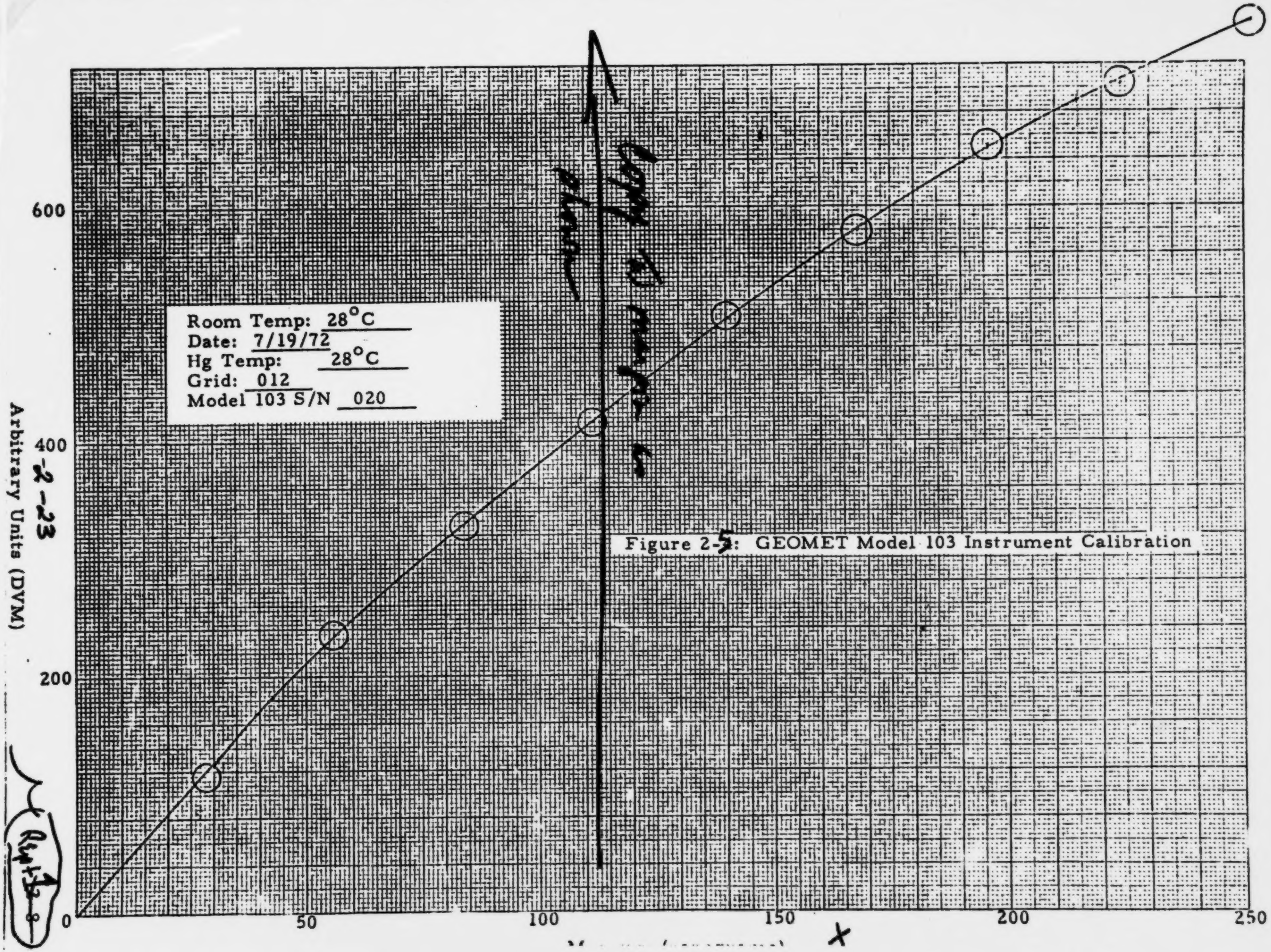


Figure 2-5: GEOMET Model 103 Instrument Calibration

Room Temp: 28°C
Date: 7/19/72
Hg Temp: 28°C
Grid: 012
Model 103 S/N 020

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air sample to the Model 103. Following preliminary mercury challenge testing, it was found that the sensitivity of the Model 103 required dilution of the incoming sampled air to provide accurate and reproducible readings at the higher mercury challenge levels. A dilution step was added to the system and calibrated to determine validity of incoming mercury challenge levels, background and overall test results when the diluter was in use. The sampling train then in use is depicted in Figure 2-6.

An interim Hi-Vol sampling arrangement was instituted wherein the Model 103 probe was positioned within the collection plenum to monitor collection efficiency of the absorbents during each challenge run. A critical orifice, with pick-up connections for a Magnehelic gage, was installed for monitoring air flow through the stacked canister system. The details of this interim air sample monitoring mode are provided in Figures 2-7 and 2-8. This new method allowed positive control over air volumes passing through the canister, which was critical to absorbent collection efficiency data calculations. It was felt, however, that the Model 103 should monitor the mercury challenge level rather than possible break-through (loss of collection efficiency) in the absorbent canister system. This decision was supported by the fact that no break-through had occurred in any of the high level mercury challenge runs. Therefore, the Model 103 canister sample output probe was sealed and reinstalled on the Hi-Vol sampler exhaust downstream of the blower housing. This final change to the collection plenum is shown in Figure 2-9.

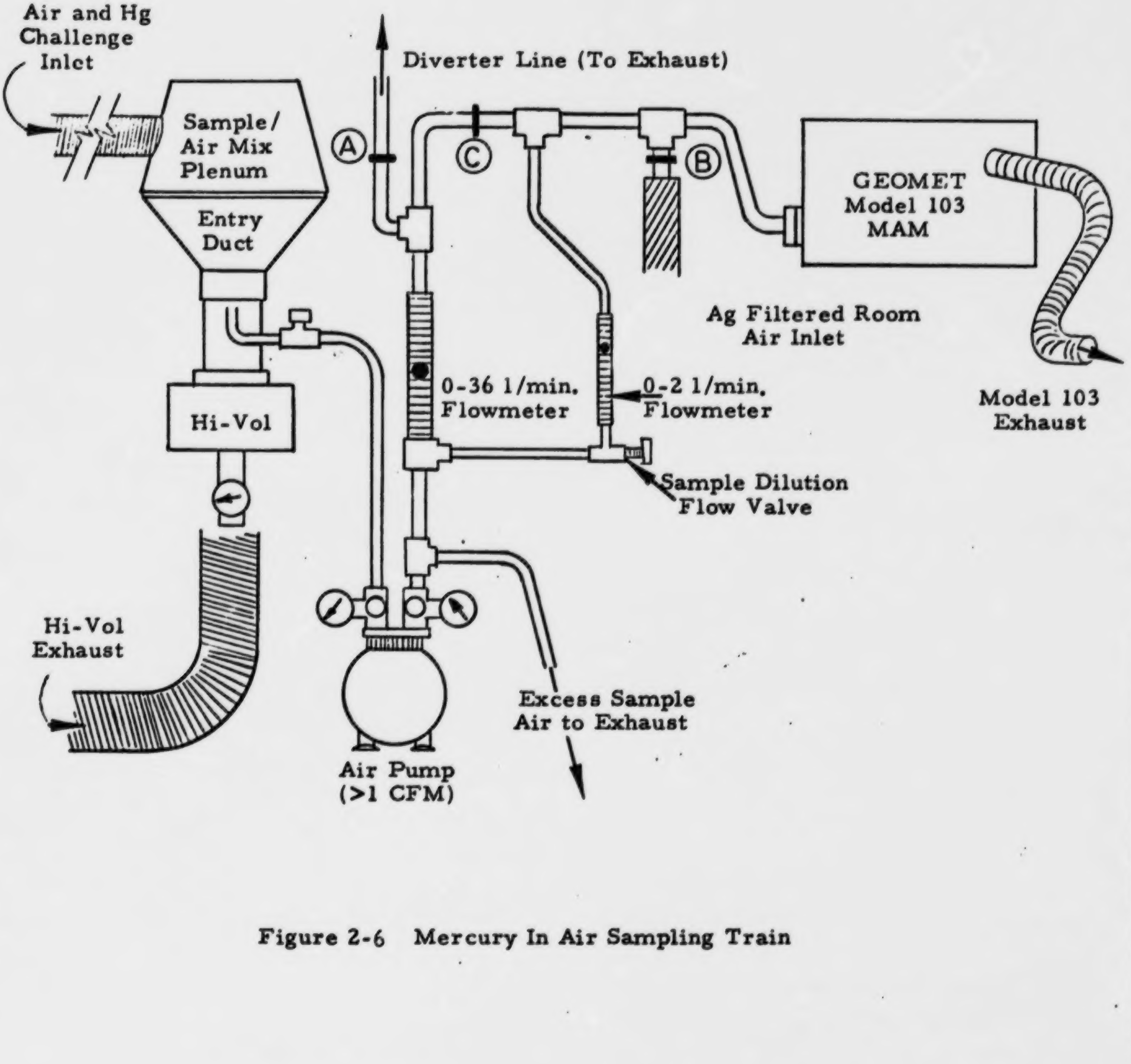

Air and Hg Challenge Inlet
Diverter Line (To Exhaust)
Sample/ Air Mix Plenum
A
C
B
Entry Duct
GEOMET Model 103 MAM
Ag Filtered Room Air Inlet
Hi-Vol
0-36 l/min. Flowmeter
0-2 l/min. Flowmeter
Model 103 Exhaust
Sample Dilution Flow Valve
Hi-Vol Exhaust
Excess Sample Air to Exhaust
Air Pump (>1 CFM)

Figure 2-6 Mercury In Air Sampling Train

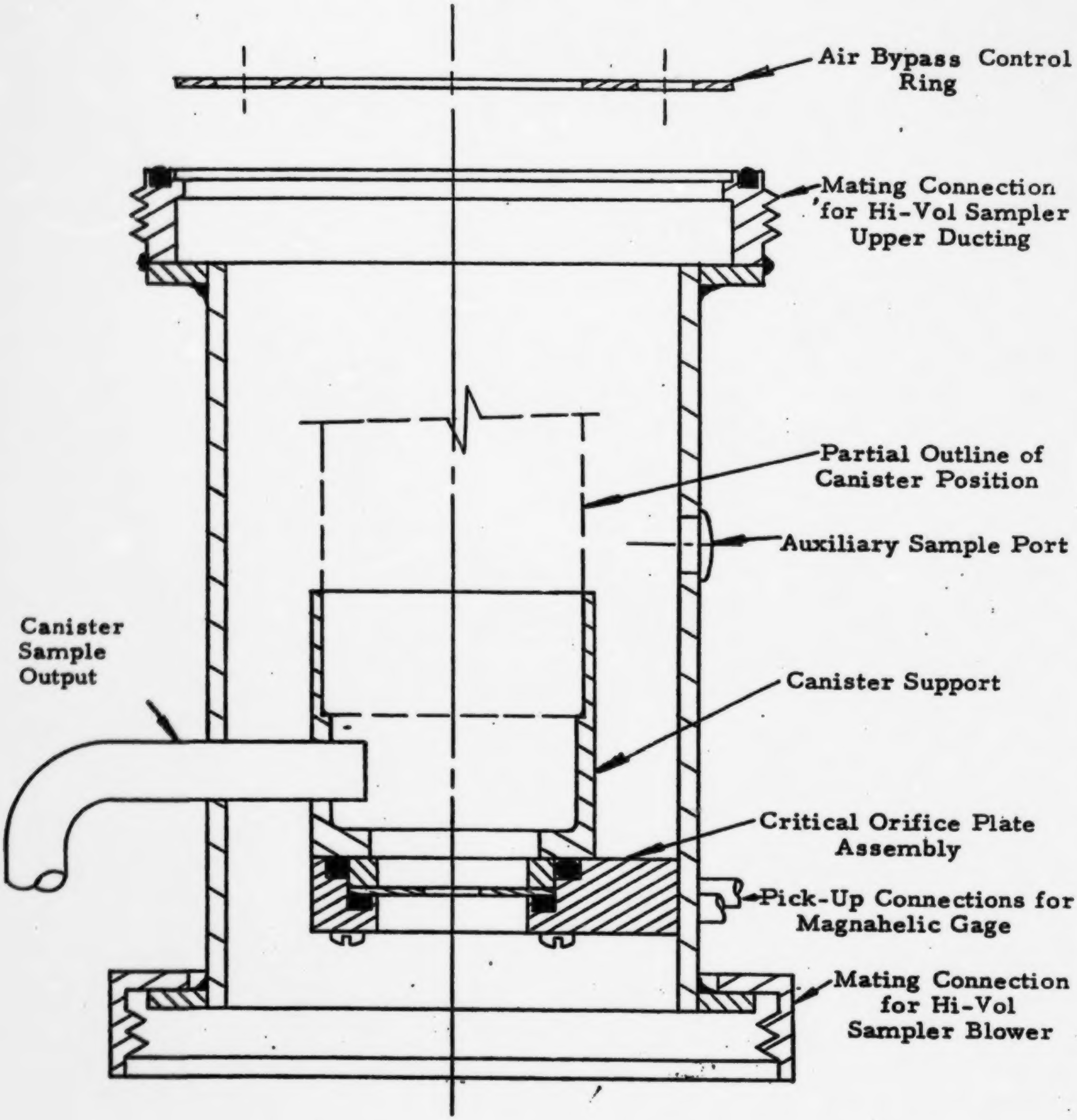

Air Bypass Control Ring
Mating Connection for Hi-Vol Sampler Upper Ducting
Partial Outline of Canister Position
Auxiliary Sample Port
Canister Sample Output
Canister Support
Critical Orifice Plate Assembly
Pick-Up Connections for Magnahelic Gage
Mating Connection for Hi-Vol Sampler Blower

Figure 2-7

Canister Holder and Sampling Plenum for Hi-Vol Sampler

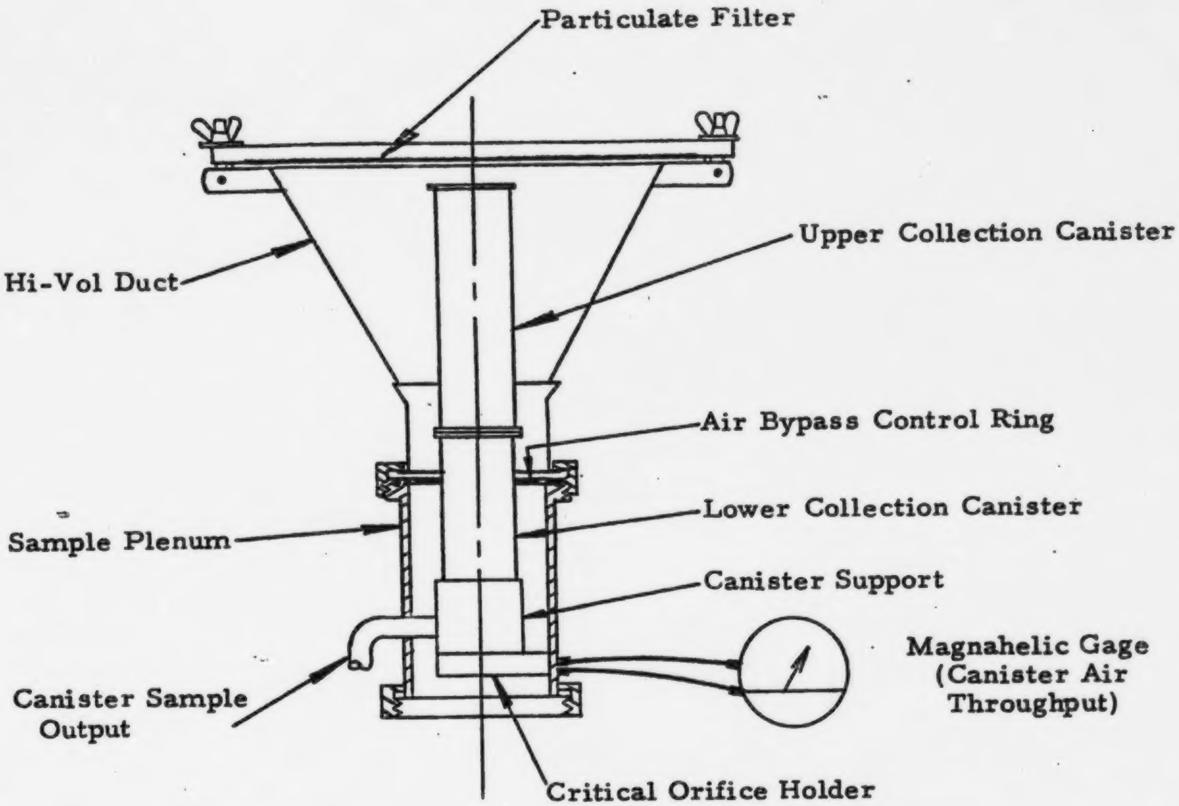
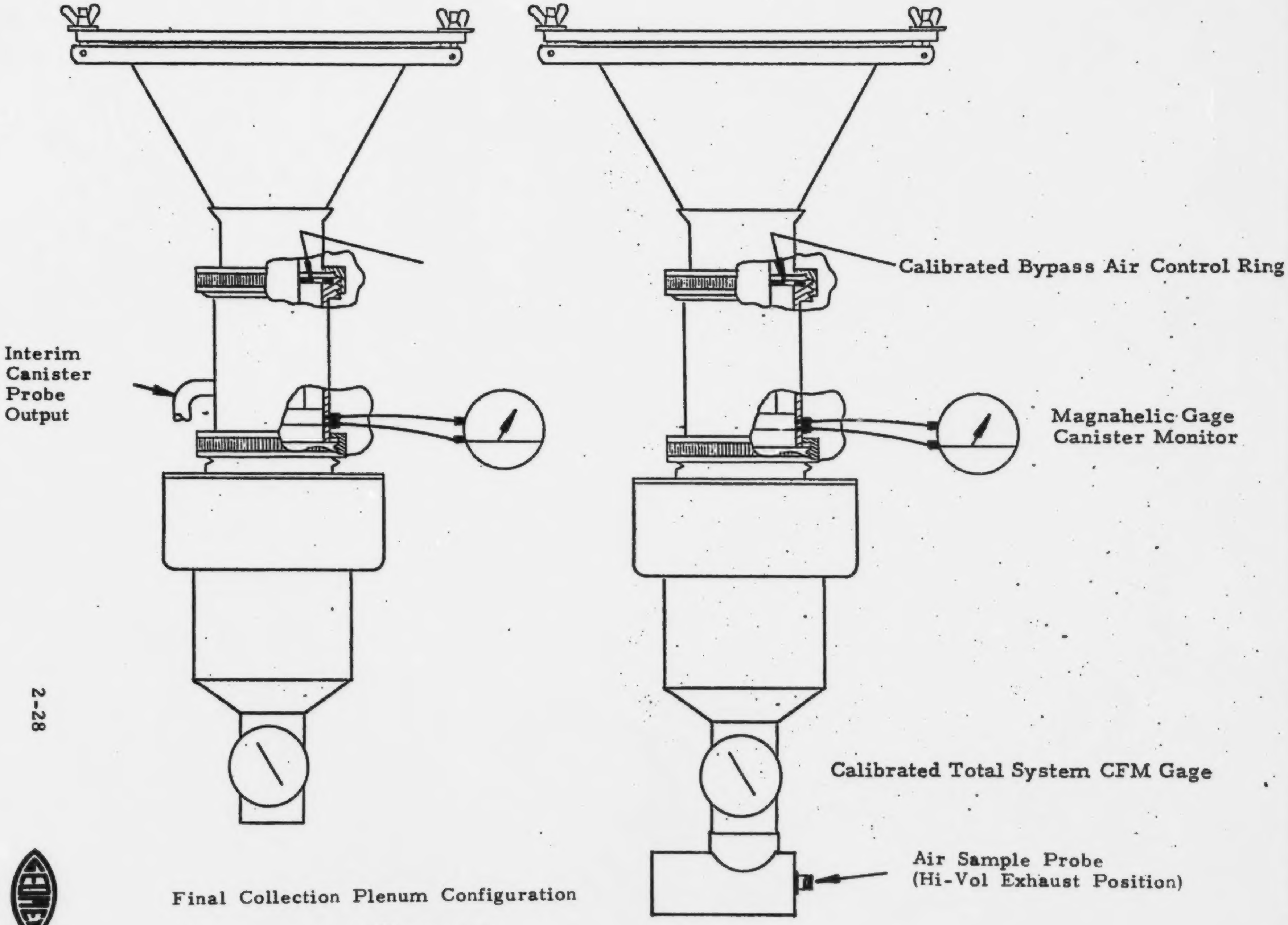

Particulate Filter
Upper Collection Canister
Hi-Vol Duct
Air Bypass Control Ring
Lower Collection Canister
Sample Plenum
Canister Support
Magnahelic Gage (Canister Air Throughput)
Canister Sample Output
Critical Orifice Holder

Figure 2-8

Collection Canister/Hi-Vol Sampler Interface Configuration


Interim
Canister
Probe
Output
Calibrated Bypass Air Control Ring
Magnahelic Gage
Canister Monitor
Calibrated Total System CFM Gage
Air Sample Probe
(Hi-Vol Exhaust Position)

Final Collection Plenum Configuration

Figure 2-9

During the latter part of this program, GEOMET decided to miniaturize the entire Air Train Challenge Apparatus described above (Ref. Figure 2-6). Flowmeters measuring in the ranges of 0-5 and 0-25 lpm were purchased and the entire dilution system was mounted on the side of the Hi-Vol sampler shelter. All tubing connections, including a column for silver-treated alumina pellets for mercury-free dilution air, were converted to 316 stainless steel. A positive pressure diaphragm pump was installed for precise control of sample air from the Hi-Vol to the Model 103. This configuration has created a simple and compact test arrangement which allows a more convenient work package and portability of the test system. The final apparatus is depicted in Figure 2-10.

2.5.1 GEOMET Model 103 - Mercury Air Monitor

2.5.1.1 General Description

The experimental measurements of mercury in air streams utilized for controlled test purposes were obtained, in part, with the GEOMET Model 103 Mercury Air Monitor. A schematic diagram of the M103 is shown in Figure 2-11. This device was set to produce an analysis every 3-6 minutes depending on the level of mercury in the gas. For organic mercury compounds the Model 103 was coupled with a Catalytic Converter (Module 109) which reduced all compounds to elemental mercury for analysis (Figure 2-12).

The Model 103 operates on the principle illustrated in Figure 2-11. Air is drawn into the instrument at pre-selectable flow rates and sampling time cycles across a silver collection grid which

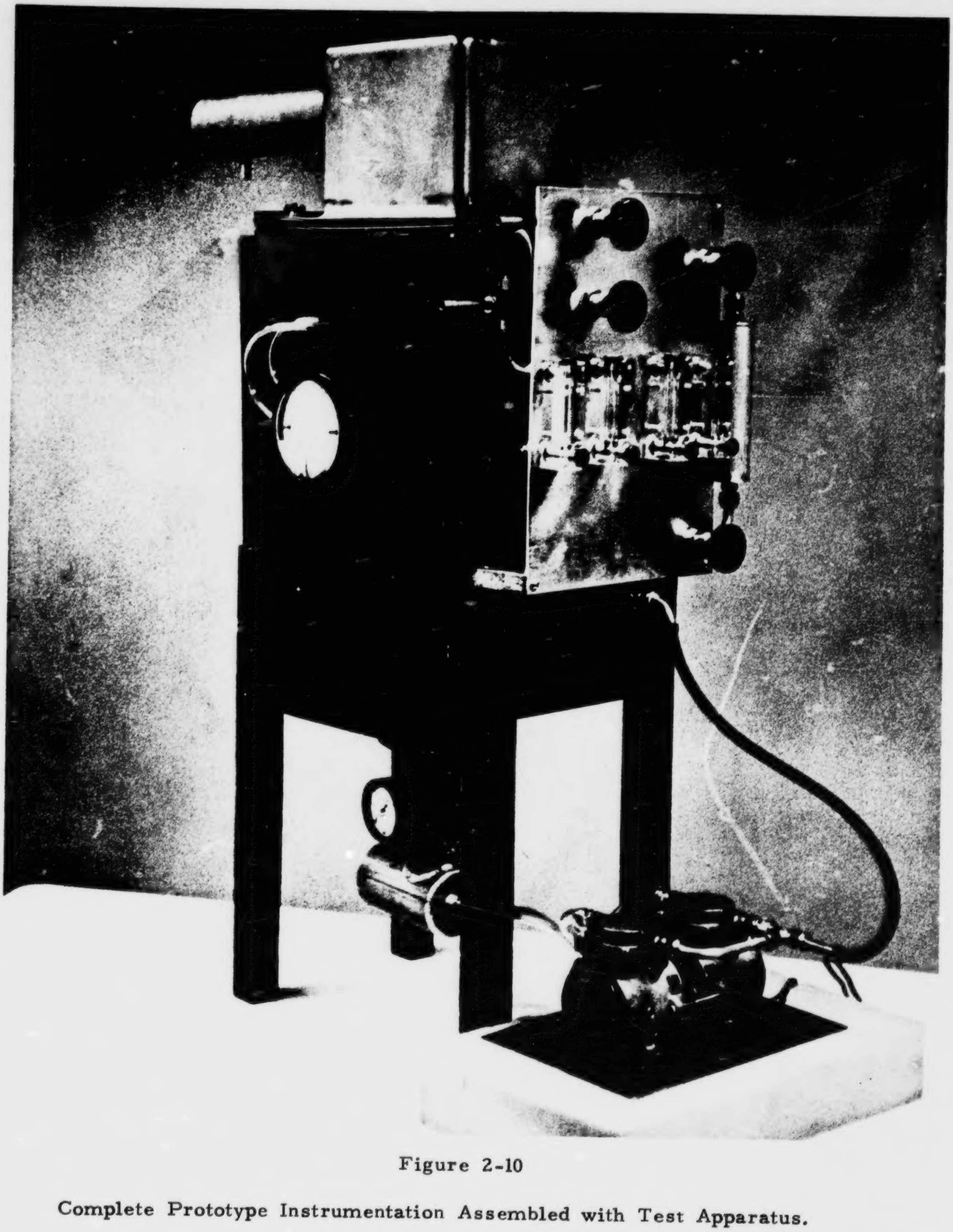


Figure 2-10

Complete Prototype Instrumentation Assembled with Test Apparatus.

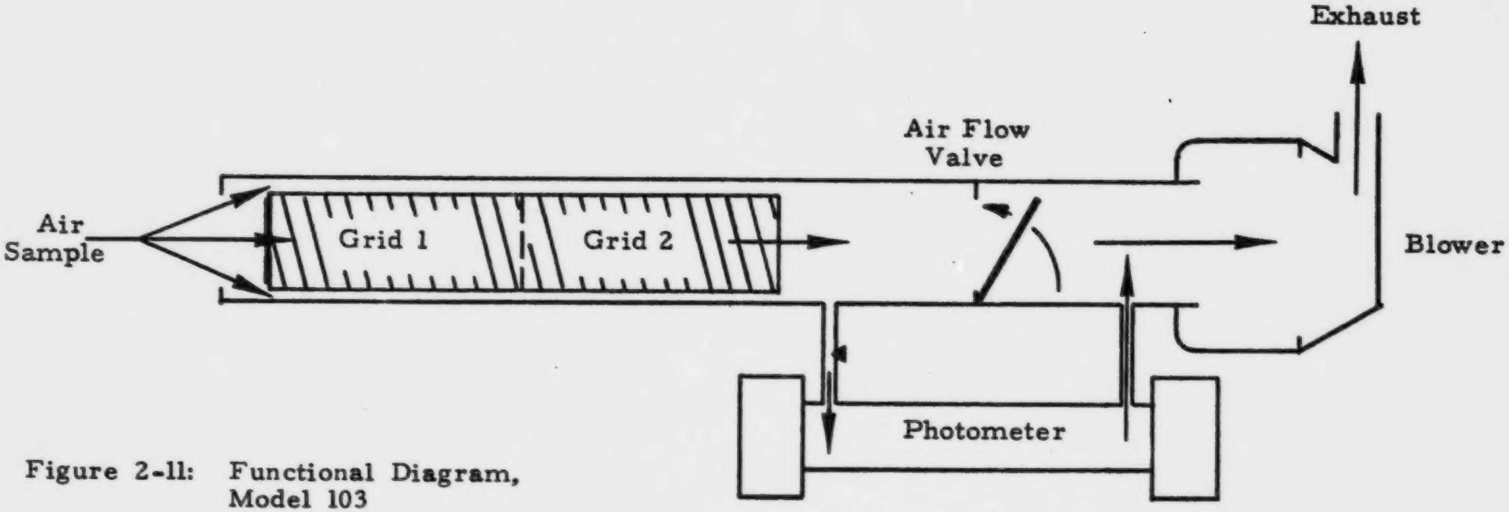

Exhaust
Air Flow Valve
Air Sample
Grid 1
Grid 2
Blower
Photometer

Figure 2-11: Functional Diagram, Model 103

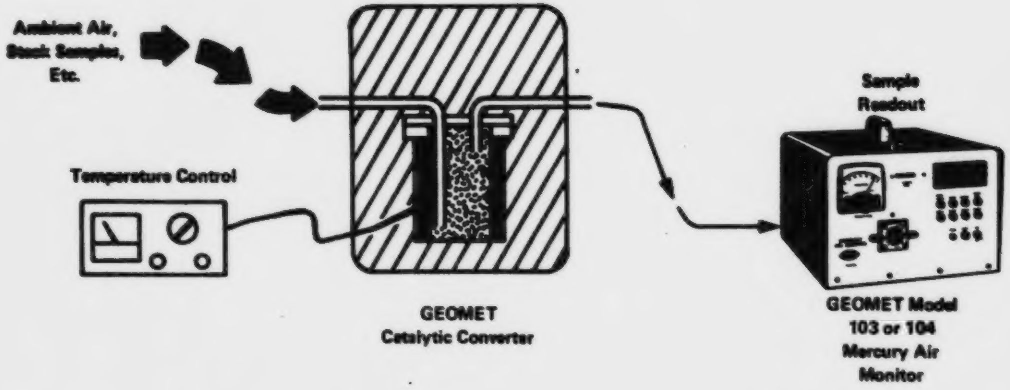

Ambient Air, Stack Samples, Etc.
Temperature Control
GEOMET Catalytic Converter
Sample Readout
GEOMET Model 103 or 104 Mercury Air Monitor

Figure 2-12: Model 103 with Catalytic Converter

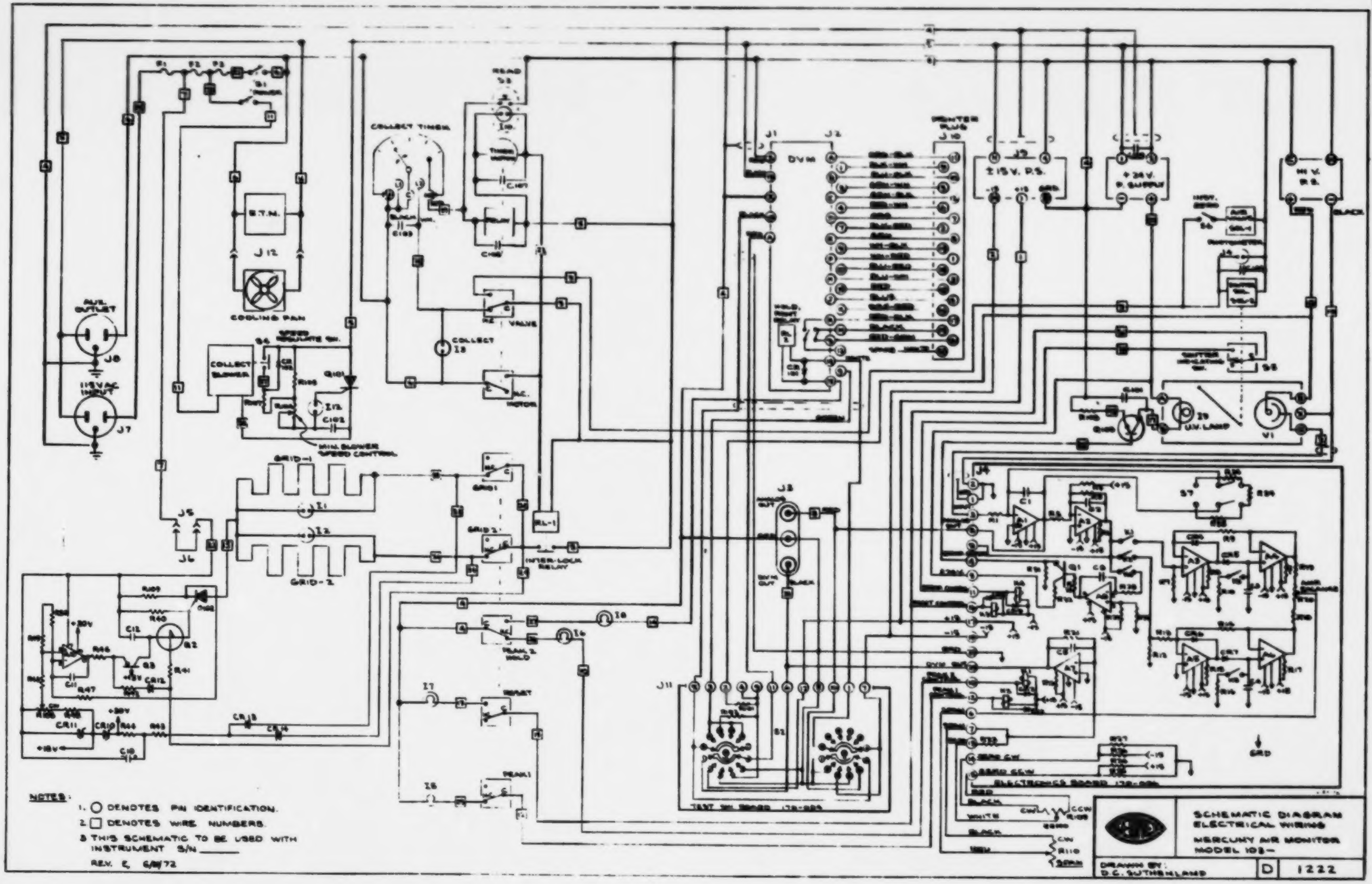
serves to concentrate the sample. Readout is achieved by use of sequential heating of the two electrically independent grids sections. During heating of the first section by direct passage of electrical current through the silver wire, collected mercury is shifted to the rear or second section. Adsorbed impurities or potential interferences, which are not strongly bound to silver, pass into the photometer for quantitation. The signal, if any, is stored electronically for subtraction from the signal resulting from heating the second grid section. Heating of the second section releases the collected mercury sample plus any collected interferences into the photometer for quantitation. The readout procedure is automatically controlled, it requires 70 seconds. The entire readout cycle requires 2.0 minutes. At 70 seconds, the corrected peak signal voltage is displayed on a digital voltmeter. Connections are provided for simultaneous use of a printer or strip chart recorder. The collection, readout and data presentation cycle is adjustable for continuous air monitoring over long periods of time.

The schematic diagram of the electrical system is detailed in Figure 2-13.

2.5.1.2 Improvements to Model 103

Field testing of the original Model 103 showed two weaknesses: (1) instability in the UV source output was not detectable. This caused poor reproducibility at low mercury levels, and required constant vigilance to avoid variations from calibrated performance, and (2) line power variations resulted in damage to the grid.

Reproduced from best available copy.


J1
J2
DVM
PRINTER PLUG
J10
±15 V. P.S.
+24 V. P. SUPPLY
HI V. P.S.
U.V. LAMP
V1
J3
J11
J12
B.T.M.
COOLING FAN
AUX. OUTLET
J8
115VAC INPUT
J7
COLLECT BLOWER
MIN. BLOWER SPEED CONTROL
COLLECT TIMER
READ
COLLECT
VALVE
A.C. MOTOR
GRID-1
GRID-2
GRID 1
GRID 2
INTER-LOCK RELAY
RL-1
J5
J6
PEAK 2 HOLD
RESET
PEAK 1
CR11
CR10
C10
+18V
GRD
-15
+15
BLACK
WHITE

NOTES:

1. ○ DENOTES PIN IDENTIFICATION.
2. ☐ DENOTES WIRE NUMBERS.
3. THIS SCHEMATIC TO BE USED WITH INSTRUMENT S/N ______

REV. E, 6/8/72

SCHEMATIC DIAGRAM
ELECTRICAL WIRING
MERCURY AIR MONITOR
MODEL 103—

DRAWN BY:
D.C. SUTHERLAND

D 1222

Figure 2-13

Both problems have been eliminated by redesign: (1) an automatic lamp intensity control has been added, and (2) an SCR current control has been added to both grid circuits. The circuitry involved is shown schematically in Figures 2-14 and 2-15. Other new features include (a) a shutter, (b) Instrument Zero Adjustment and (c) Span Adjustment.

2.5.1.3 Details of Changed Features

Automated UV Lamp Current Control

An automated UV lamp current control system is now included in the Model 03 Mercury Air Monitor. This "loop" system obviates the necessity for manual adjustment of the UV lamp, and offers stable lamp conditions for the life of the bulb. This system contains two functions:

1. "Sampling" error voltage control,
2. "Hold" error voltage control.

The purpose of the "Sampling" error voltage control is to maintain the UV lamp intensity, measured at the photodiode, at a constant level prior to the peak reading cycle. The "Hold" error voltage control maintains a fixed, but non-controlling, voltage to the lamp during peak reading cycles.

On instrument start-up, the Model 103 Mercury Air Monitor is set up ready for air sampling, the power switch is actuated to the "ON" position and the instrument operation observed. The operator then depresses the manual READ pushbutton. The purpose of this action is to initially start the UV lamp, and therefore actuate the

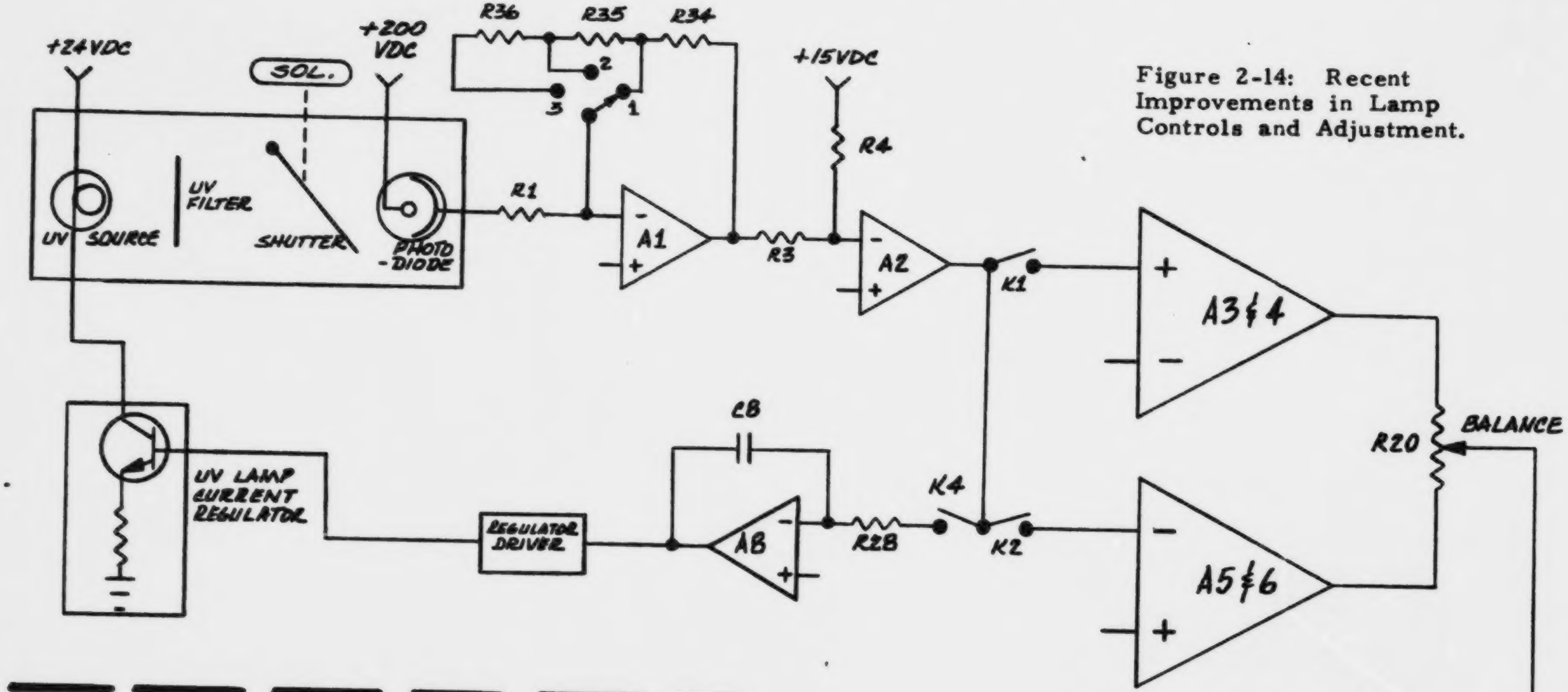

+24VDC
SOL.
+200 VDC
R36
R35
R34
2
3
1
+15VDC
R4
UV SOURCE
UV FILTER
SHUTTER
PHOTO -DIODE
R1
A1
R3
A2
K1
A3 & 4
UV LAMP CURRENT REGULATOR
C8
REGULATOR DRIVER
A8
R28
K4
K2
A5 & 6
R20
BALANCE

Figure 2-14: Recent Improvements in Lamp Controls and Adjustment.

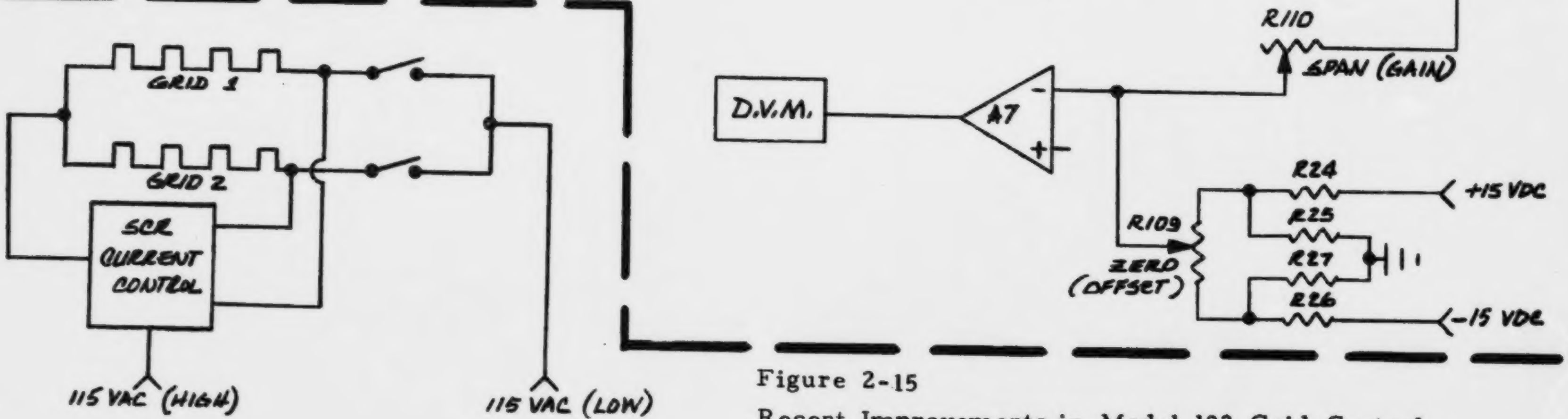

GRID 1
GRID 2
SCR CURRENT CONTROL
115 VAC (HIGH)
115 VAC (LOW)
R110
SPAN (GAIN)
D.V.M.
A7
R24
+15 VDC
R25
R27
R26
R109
ZERO (OFFSET)
-15 VDC

Figure 2-15

Recent Improvements in Model 103 Grid Control Circuitry.

automated UV lamp current control system and instrument readout cycle. The DVM readouts for the first fifteen minutes are considered invalid due to the need for UV lamp stabilization.

Once the lamp is actuated by the first READ cycle, the lamp voltage is automatically controlled. Current fed to the lamp is monitored by a transistorized current leveling circuit. Should the output voltage of the lamp (measured by the photodiode) vary above or below the preset value (normally 9.85 ±0.20 volts), the current is automatically reduced or increased to maintain the desired value.

During the sample readout cycle, the control system shifts to the "Hold" error voltage control system. Coincidental to the sample signal being measured by the peak detectors, the variable voltage control is by-passed and the lamp is held at a constant level value and not adjusted during the peak reading cycles. This provides a constant unchanging baseline voltage over which the sample value is superimposed.

Following the sample readout, the lamp system returns to the "Sampling" error voltage control mode. Here, the error voltage control is used to sustain the UV lamp temperature and activation concurrently with the use of a mechanical shutter positioned in front of the photodiode. This condition maintains the lamp in a ready state, and the shutter extends the life of the photodiode by cutting off UV radiation from the lamp.

The shutter, which has been installed in front of the photodiode, remains in the closed position during the sample collection cycle. The shutter blade interrupts the passage of light from the UV lamp to the photodiode. During the "Read" cycle the shutter is lifted out of the light path and allows the full output of the lamp to reach the photodiode. This action occurs prior to the entry of the air sample into the photometer. The shutter is timed to close following the readout cycle, again protecting the photodiode. The shutter has increased the photodiode life to something greater than 30,000 readout cycles.

Instrument Zero Adjustment

This adjustment provides a method of evaluating any electronic "noise" within the Mercury Air Monitor, establishes that level of noise and provides adjustment to compensate for that signal level in the final readout value. This adjustment can be performed with any amount of mercury in the sampled air passing through the system.

In operation, the Instrument Check Switch (located on the rear instrument panel) is moved from the "Normal" position, UP to the "Zero Chk" position. The "Read" button, on the front panel, is then pushed to activate the readout cycle. During the Peak 2 portion of the cycle, the Zero Knob (located on the front instrument panel) is adjusted to maintain a reading of +000, or any other desired positive value. At this point, the Instrument Check Switch should be returned to the "Normal" position.

This action has now calibrated the instrument for full electronic zero, including the collection grid firing cycle. The Instrument Zero Adjustment should be made following any replacement of collection grid assemblies, and as often as operational use requires.

Span Adjustment

The Span Adjustment is used to calibrate the Mercury Air Monitor upper limit readout. The instrument zero point is adjusted with the Instrument Zero Adjustment, and the Span Adjustment is then necessary for full instrument calibration. The span is adjusted prior to shipment and generally is not readjusted by the instrument user. A procedure for changing the span is included in the Operations Manual. An additional use of the Span Adjustment is for matching the sensitivities of two or more Mercury Air Monitors being concurrently utilized for air sampling so that the same mercury vapor level injected into all instruments yield the same final reading on all instruments.

Grid Temperature Control

It has been observed that full line voltage is not required by the grids for desorption of collected mercury. By installation of SCR current controls, the grid current is limited to a maximum of 15 amperes. Previously an upper limit of approximately 30 amperes was obtained. Chronologically, since this limitation was imposed on the silver grids, no single grid failure has been noted in more than 4000 hours of operation.

2.6 MERCURY ABSORBENT DEVELOPMENT

This section of the program Final Report is devoted to discussions of the development of unique and efficient collection materials for the three forms of mercury - particulate, elemental and organic. Rationale for approaches utilized for each material type is provided.

2.6.1 Selection of Adsorbents

The following remarks form the rationale for selection of the silver on alumina adsorbent utilized as a major candidate for the collection medium for elemental mercury and for charcoal as the collector of organic mercury:

Adsorption of elemental mercury and its compounds from the gas phase onto solids is largely a surface phenomenon. The amount of adsorbate, its rate of adsorption and the efficiency of mercury extraction from the gas are dependent on the specific surface and the total surface of the adsorbent which is presented in the process. Other factors which control the removal capacity include the nature of the adsorbent and adsorbate, the geometrical state of the adsorbent, the temperature and velocity of the air, the concentration of the mercury-containing gas, effects of other gases in the stream and the proportion of the adsorbent surface covered as the adsorbate collides with the surface. In general, the adsorbent bed operates efficiently until the total capacity of the bed is approached.

Desirable adsorbent properties include:

(a) Capacity: 15-30% of adsorbent weight,

(b) Low resistance to gas flow,

(c) Inertness,

(d) Resistance to deterioration during use,

(e) Regenerability, and

(f) Provide ready recovery of adsorbate for analysis.

The adsorption of elemental mercury is most efficiently achieved on the noble elements, gold, silver and platinum where the chemisorptive mechanism resembles amalgamation. Selection among these metals has largely been made on the basis of economics: silver is vastly less expensive. Thin layers of gold on supports have been utilized (References 1-7) but regenerability of very thin films is usually poor. Also, both gold and platinum tend to hold onto small portions of mercury tenaciously if present in reasonable mass. The recovery of elemental mercury from silver is completed at relatively low temperatures.

Reference 1: S.H. Williston and M.H. Morris; U.S. Patent 3,173,016 (1965); and S.H. Williston; U.S. Patent 3,178,572 (1965)

Reference 2: W.W. Vaughn and J.H. McCarthy; U.S. Geological Survey Prof. Paper pp D123-127 (1964).

Reference 3: S.H. Williston; Jour. of Geophys. Res, 73, 7051 (1968).

Reference 4: D.H. Anderson, J.H. Evans, J.J. Murphy and W.W. White; Anal. Chem. 43, 1511 (1971).

Reference 5: L.M. Azzaria; Canadian Geolog. Survey Paper No. 66-54, pp 13-26 (1967).

Reference 6: J.J. McNerney and P.R. Buseck; Science 178, (10 November, 1972) 611 (1972).

Reference 7: NTIS Report No. PB-210 817. TraDet, Inc. Columbus, Ohio (1972).

Typical surface areas for various adsorbents and adsorbent supports are shown in the following table:

Support	Specific Surface Area (m^2/g)
Pyrex Wool (Corning, No. 3940	0.27*
Glass Fiber (Fiberglas, various)	0.04 - 0.16
Activated Alumina (Harshaw, Al-0104T)	80 - 100
Activated Alumina (Alcoa)	100 - 350
Activated Charcoal (Barnebey-Cheney, TCA)	1000
Activated Carbon (Darco)	612 - 1190
Activated Carbon (PCC)	1100
Activated Carbon (Nuchar)	750
Silica Gel (Davison)	400 - 800
Silica Gel (Monsanto)	520
Attapulgus Clays	~120
Bentonite (Filtrol)	280
Fuller's Earth (Floridin)	124
Silica-Alumina (various)	500 - 600
Activated Magnesia (Westvaco)	30 - 230

Ranges are presented where more than one product is available. Surface areas may be varied by modification of preparational techniques. For example, activated carbon may be made with a 10 m^2/g surface area by heating at 2750°C. Steam and heat treatments may be used to reduce the surface areas of all silicate structures.

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*Calculated on basis of average fiber diameter and density.

On this basis the following data show the surface areas utilizable in the collection canisters (160 ml) with normal packing:

Support	Specific Surface Area (m^2/g)	Average Packing Density (g/ml)	Wt. in Canister (140 ml)(g)	Total Surface Area (m^2)	Ratio
Pyrex Wool	0.27	0.065	9.1	2.5	1.0
Alumina (Harshaw)	100	1.09	160	16,000	6,400
Activated Carbon (Barneby-Cheney, TCA)	1000	0.54	75	75,000	30,000

These data show that utilization of a nonporous solid such as pyrex wool will minimize the total surface available for adsorption of mercury. (With careful packing the glass fiber content of a canister might be doubled.) The useful surface with alumina is about 6,400 times greater than that obtained by pyrex wool; activated carbon offers ~ 30,000-fold more surface.

While the useful surface of the supports is reduced after deposition of silver, particularly at the relatively high levels used in these collection experiments, the Ag/Al_2O_3 preparations employed still offer approximately three (3) orders of magnitude more surface than would glass wool preparations in the same volume. Charcoal, used for collection of the organic mercury compounds is one of the highest surface area adsorbents in common usage.

The objective of the foregoing comparison is to indicate the advantages accruing by use of a Ag/Al_2O_3 preparation for collection of elemental mercury. It meets most of the criteria indicated in the initial paragraph of this section: good capacity, low resistance to gas flow, inertness, resistance to deterioration during use, regenerability, easy recovery of mercury for analysis and relatively economical initial costs. Other adsorbents apparently do not meet all of these criteria as conveniently. However, several other compositions should be examined in order to obtain a maximal collection technique. Two in particular, gold and silver on pyrex wool should be tested further. However, in order to overcome the obvious advantages of silver on a porous support, these would have to be at least 1000 times more efficient in capture of mercury vapor (atoms) than is silver on alumina.

2.6.2 Particulate Mercury Collector

During the progress of this program, GEOMET has utilized the standard glass fiber particulate filters normally employed in conjunction with Hi-Vol samplers. These Reeve Angel 934 AH glass fiber filters were used exclusively and gave no evidence of collected sample loss with proper handling. A unique method was devised for analysis of the mercury collected on these filters (See Section 2.8.2.), and no problems with the filters were evident in the recovery/analysis method. It is assumed that any glass fiber filter, with the same basic specifications, may be utilized to collect mercury and be amenable to the recovery/analysis method.

2.6.3 Elemental Mercury Adsorbents

At the onset of the program, GEOMET decided to manufacture trial lots of silver-treated alumina in order to evaluate the feasibility of this adsorbent type as a collector for only elemental mercury.

Several preliminary batches of silver-treated alumina were prepared with $AgNO_3$ from samples of 0.125 alumina pellets (Harshaw, Surface Area: 80-100 m^2/g), and 4-8 and 8-16 mesh alumina granules (irregular shapes). These batches contained 2-5% Ag by weight. Volumes of these prototype mercury collection substrates were tested in tubular canisters (3" diameter) placed inside the Hi-Vol sample ducting upstream of the Hi-Vol blower motor. Challenges utilized the system described above.

Collection Substrate	Bed Depth	Challenge Level, μg Hg/M^3	Collection Eff., %
0.125 Alumina Pellets	3.0" (300 gm)	44	64%
4-8 Mesh Alumina Granules	3.0" (350 gm)	48	96%
0.125 Alumina Pellets	5.0" (710 gm)	230	>99%
0.125 Alumina Pellets	6.0" (855 gm)	230	>99.7%
0.125 Alumina Pellets	7.0" (1,000 gm)	230	>99.99%

These test results indicated that the pellets were very efficient in collecting mercury vapor especially in bed depths of 7.0 inches even at extremely high mercury challenge levels. Also, the Hi-Vol sampler maintained >20 CFM air through-put at this maximum bed depth. Quantities of Ag-treated 8-14 mesh alumina were tested early in the reporting period, but did not give the desired results due to (1) decreased air through-put through the Hi-Vol sampler (<20 CFM), and (2) lower collection efficiencies than the 4-8 mesh alumina granules.

Twenty-four (24) hour collection efficiency runs were made on several GEOMET-prepared batches of one-eighth inch alumina pellets treated with silver. For example, one batch contained silver coating averaging 3.5% by weight. This 1,000 gm. pellet batch was challenged with a constant "medium" level of mercury (3.3 $\mu g/M^3$) for 24 hours. The collection efficiency of this material ranged from an initial value of 99.3% to a low of 94.5% at the conclusion of the run. The result of this test was compared to similar tests run on pelletized alumina coated with $\sim$6% silver by weight. Eight hundred gm. of this material was also subjected to a 24-hour test with a challenge level of mercury vapor at 3.3 $\mu g/M^3$. The same 800 gm. batch was then challenged with a "high" level stream of elemental mercury vapor at a constant concentration of 100 $\mu g/M^3$. This latter level is stipulated in the Statement of Work as the maximum threshold limit for collection. This GEOMET-prepared product gave collection efficiency ranges of 99.2 to 92.6% with the 3.3 $\mu g/M^3$ challenge, and 99.9 to 99.3% with the 100 $\mu g/M^3$ mercury challenge. Figure 2-16 presents the results of these preliminary three 24-hour runs.

During the program progress, it became evident that the high efficiency of the silver-treated alumina would allow a Prototype system elemental canister size of $\sim$200 ml or 180 gm of treated pellets. Preparation of the GEOMET silver-treated adsorbent was refined until the pellets contained $\geq$12% silver by weight. Here, a 50/50 (w/v) solution of $AgNO_3$ and distilled water was applied to untreated 1/8 inch diameter by 1/8 inch long cylindrical alumina pellets (Harshaw Chemical Company, Catalyst AL-0104, Lot 61) under vacuum. Following full wetting of the alumina support, the pellets were dried overnight at 100°C. The pellets were then placed in a muffle furnace and heated at 600°C for two hours to drive off residual nitric acid. The pellets were then ready to use as adsorbent in elemental mercury collection canister. These particular pellets have shown typical collection efficiencies of 100 percent at 3.8 $\mu g/M^3$ for 24 hours and 97.1 percent at 28 $\mu g\ Hg/M^3$ for 24 hours. The preparation steps have been even further simplified to allow preparation of 1 kilo of treated pellets to be made in less than one hour. This time excludes oven drying of the $AgNO_3$ on the pellets, and subsequent firing of the pellets in the resistance furnace to remove the residual nitric acid. These preparations provide excellent collection efficiency and material balance (Ref. Section 2.9.2 and have been reclaimed for possible reuse by additional heating in a resistance furnace combined with circulated air to remove the bulk of collected mercury on the first reclaiming cycle. GEOMET

K•E 10 X 10 TO THE CENTIMETER 46 1513
10 X 25 CM MADE IN U.S.A.
KEUFFEL & ESSER CO.

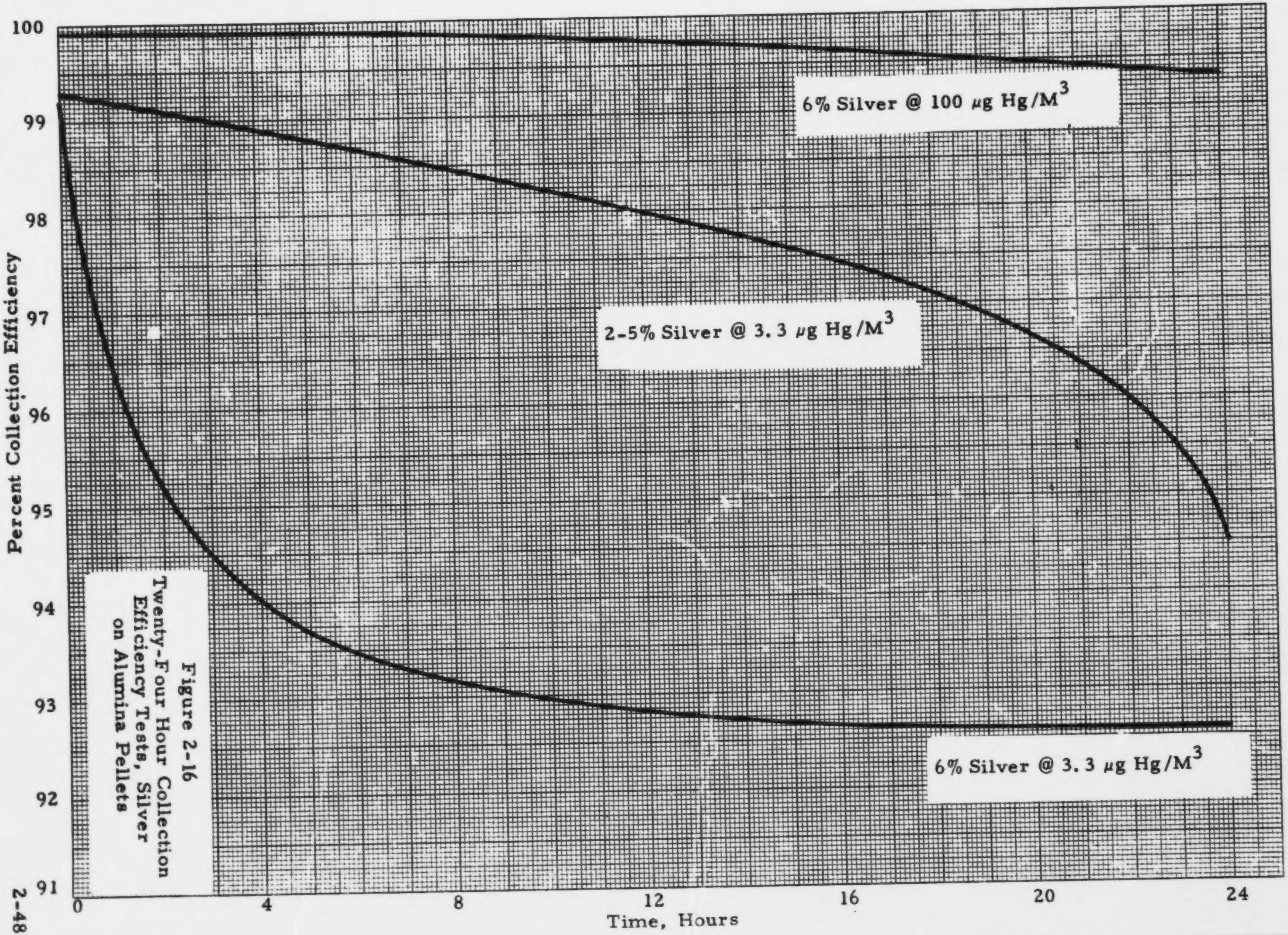


Figure 2-16
Twenty-Four Hour Collection
Efficiency Tests, Silver
on Alumina Pellets

feels that silver treatment of alumina could be converted, with some attendant processing equipment, to an economical commercial process.

To fully appreciate the rationale that GEOMET utilized in selection of silver-treated alumina pellets over more widely publicized methods, the data presented above in Section 2.6.1 (Ref. 1) should be considered in conjunction with U.S. Patent No. 3,178,572*; column 6, lines 15 through 60:

> "The mercury-absorption chambers 71 and 82 contain highly effective absorption media for the specific removal of mercury from the flowing air as compared with any other contents of the air. Useful for this purpose is glass wool having its fibers coated with pure gold. A coating of silver may also be used but this is not as desirable as gold because of its susceptibility to formation of silver sulphide under the action of hydrogen sulphide content of the air, though it may be used in sulphur-free and chlorine-free atmospheres. Other metals which are characterized by wettability by and some solubility in mercury may be used, but none has been found to be more effective than gold. The gold may be deposited on the glass wool by ordinary and convenient deposition methods, for example by merely wetting the wool with a gold salt, such as chloride, and decomposing the salt by heat for deposition of the gold. There may be used, in place of glass wool, nickel wool on which the gold is deposited in the same fashion or by precipitation by the nickel from a solution of a gold salt. A difficulty with glass wool is that glass will absorb, to some extent, mercury, and consequently the dummy chamber 94, containing the same amount of glass wool but uncoated, will be required to be satur-

---

*ULTRA-VIOLET RADIATION ABSORPTION ANALYSIS APPARATUS FOR THE DETECTION OF MERCURY VAPOR IN A GAS.
Samuel H. Williston, Los Altos, Calif., assignor to Cordero Mining Company, Palo Alto, Calif., a corporation of Nevada
Filed May 17, 1963, Ser. No. 281,088
6 Claims. (Cl. 250-43.5)

> ated to the extent of this absorption before use. In the case of nickel, however, the absorption of mercury is negligible. Other carriers of gold or silver may also be used, such as alumina, completely coated to prevent absorption of water. The general properties of the carrier should be that of a physical form to present a maximum absorbing surface of the noble metal per unit volume, reasonably low resistance to flow, adhesion to its absorbent coating and non-destructible by heat used to drive off mercury. In itself, it should be non-absorptive of mercury, or a least exhibit uniform absorption thereof. The last property may be best described by saying that the carrier should be non-wettable by mercury. It should, so as to be usable in the dummy chamber, be non-absorptive of other substances which absorb the radiation bands absorbed by mercury. The noble metal coating on the carrier should be very thin, ranging from a small fraction of a thousandth of an inch to not more than a few thousandths. The reason for the latter limitation is that if a thick coating of gold, for example, is used, the absorbed mercury will diffuse deeply thereinto and will not be driven off completely in regeneration of the absorbent by heat at moderate temperatures."

GEOMET's selection of alumina over glass wool was basically due to the gross differences in available surface area for mercury collection (Ref. Section 2.6.1, Page 2-42). The choice of silver rather than gold, as a noble metal mercury collector, markedly reduces the cost of the final alumina adsorbent. As previously reported, it appears that this product would be commercially produced easily and economically.

2.6.3.1 Commercially Available Silver-Treated Adsorbents

During the program effort, commercially available silver-treated alumina and molecular sieve material was purchased from W.R. Grace Company (Davison Chemical Division); Strem Chemicals, Incorporated; and Coast Engineering Laboratory.

The Grace product, designated as SMR 7-4215, was stated to contain 7.5% silver deposited on 4-8 mesh alumina granules. This material was tested twice. The first test was carried out in preparation for a 24-hour efficiency test i.e., it followed all standard background measurements, etc. taken on the GEOMET sampling train. A canister containing 910 gm. of this material failed, however, to remove 0.8 μgm. of mercury vapor contained in a 30 second pulse. The collection efficiency was 0% with this challenge duration and level. The Grace product was then subjected to heat treatment in a muffle furnace where it was held for two hours at 500°C. When 42 gms. of this material was subjected to the output of the GEOMET Hg vapor source (a standard screening procedure) at 42°C, it absorbed the elemental mercury vapor challenge well enough to warrant the heat treating of a 910 gm. batch of granules. This batch was prepared and entered the 24-hour testing cycle but again failed a 30 second pulse of mercury heated to 42°C (0.8 μgm.).

The Strem material, Lot 837-S, contains 11% silver by weight on alumina pellets with 0.5 M^2/gm. surface area. A 42 gm. aliquot of this material was subjected to mercury vapor at 42°C from the GEOMET Hg vapor source and collected no calculable amount of mercury. An equivalent aliquot was heat treated at about 1,000°C for one hour and then challenged by the GEOMET vapor source for 12 hours. Inasmuch as this was a lengthy screening test, no Model 103 data were collected during this period of time, during which the collection efficiency dropped from $\geq$99% to 0% and the exact time of collection efficiency loss is not known. A mass of 900 gms. of this material was then heat treated at 1,000°C and rerun at this volume and weight in a 24-hour collection test. The treatment did not enhance the collection efficiency.

GEOMET purchased 300 gms. of Silver X, Type 13X, 12-16 mesh, chromatographic grade molecular sieve material (Coast Engineering Laboratory). This compound is the result of an exchange process wherein the sodium in the Zeolite has been completely exchanged by silver. The sieve was subjected to bench-scale mercury collection tests, and subsequently two 100 gram batches were included in total system collection efficiency tests (Test data table Section 2.9.1). Both runs gave very poor results, insofar as the material balance was no greater than 57.7%. Some difficulty was found in removal of the collected mercury with the recovery/analysis technique. It is felt that this was due, in part, to the initial recovery furnace configuration (Reference Section 2.7.4. Also, the internal structure of the Silver X may preclude easy recovery of absorbed mercury (Ref. Patent 3,178,572 above). Due to the short duration of the program, and the excellent results of the GEOMET-prepared silver-treated alumina, no further effort was expended on this particuular mercury adsorbent. It does, however, have considerable promise as a collection medium.

2.6.4 Organic Mercury Absorbents

Many tests were carried out to evaluate the collection capability and recovery characteristics of commercially available charcoals to be used for organic mercury vapor collection. Three types of charcoals were evaluated: (1) Silver Impregnated (10% Ag by wt.) Charcoal, lot 841S, 4-8 mesh, Strem Chemicals, Inc., (2) Sulfur-Treated (12%) Charcoal, CB-1786, 4-8 mesh, Barnebey-Cheney, and (3) Activated Charcoal, TCA, 4-8 mesh, Barnebey-Cheney. All three charcoals were initially bench-tested for mercury collection and recovery characteristics. The Strem material collection efficiency was approximately 35% that of either the sulfur treated or TCA activated charcoals. The Strem material was therefore eliminated from further consideration as a program candidate.

The Sulfur-Treated (12%) Charcoal (CB-1786) collected within approximately 85% of the TCA activated charcoal. The results of tests for evaluation of mercury recovery indicated problems with the utilization of sulfur-treated product, however. This charcoal was found to liberate an apparent heavy hydrocarbon-like material when heated in the mercury recovery resistance furnace. The impurity appeared to come out of the vapor state, downstream of the furnace, and it coated the interior surfaces of the transport tubing ahead of the iodine monochloride collection bubblers. This residue trapped some of the mercury vapor being transported to the bubblers, and therefore severely affected the mercury recovery and material balance results of these tests. This phenomenon justified the elimination of the sulfur-treated charcoal from further consideration as a useful organic mercury collector.

GEOMET then utilized 75 gm aliquots of Barnebey-Cheney TCA activated charcoal for all organic mercury collections. This absorbent volume, without any special loading or packing procedures, occupies approximately the same volume (~160 ml) in the proposed Prototype collection canister as does the 180 gms of silver-treated pellets utilized for elemental mercury collection. The collection efficiency and mercury recovery from the TCA charcoal was excellent, and was reported as the final GEOMET choice for organic mercury absorbent utilized in the Prototype instrumentation.

2.7 PROTOTYPE COLLECTION SYSTEM

The various developmental details of the total Prototype system are discussed throughout this program Final Report. The details of the final configuration are included here, including fabrication sketches of each component.

2.7.1 General

The design of the Prototype system has been kept uncomplicated to offer minimum development costs and maximum operational characteristics. The materials of construction, e.g. 6061T6 aluminum, 316 stainless steel, PVC plastics, etc., are free from mercury upon purchase. These materials are utilized in areas to obviate the unnecessary contamination by mercury forms, and where the possibility of low mercury background might occur, methods have been devised for readily cleaning all component surfaces.

The Prototype system consists of 1) a Hi-Vol sampler collection plenum, 2) two absorbent canisters, 3) an air through-put control ring, 4) absorbent materials (Ref. Section 2.6), 5) an air metering system, and 6) a Unico 550 Turbine Jet High-Volume Sampler with a wooden shelter.

Attendant testing devices include an elemental mercury vapor source (Ref. Section 2.4.2), a GEOMET Model 103 Mercury Air Monitor (and a M109 Catalytic Converter Module) for tracking mercury challenge levels, and a Processing System for the recovery and quantitative analysis of mercury from particulate, elemental and compound vapors, respectively. (Reference Section 2.8).

2.7.2 Hi-Vol Collection Plenum

The major component of the Prototype collection system is the Collection Plenum which is inserted between the Hi-Vol blower motor housing and the adaptor ducting. The plenum is attached with standard threaded screw rings which fit commercially available Hi-Vol samplers. Without the canisters, the plenum contains canister supports, a removable calibrated air bypass control ring, and a calibrated orifice plate for the monitoring of canister air through-put. There are feed-through connections, from the orifice plate, through the plenum wall for the attachment of a Magnehelic pressure gage. A drawing of the assembled system is provided in Fig. 2-17, and a photograph of the unit in Fig. 2-18. Fabrication drawings are provided in Figures 2-19 through 2-27.

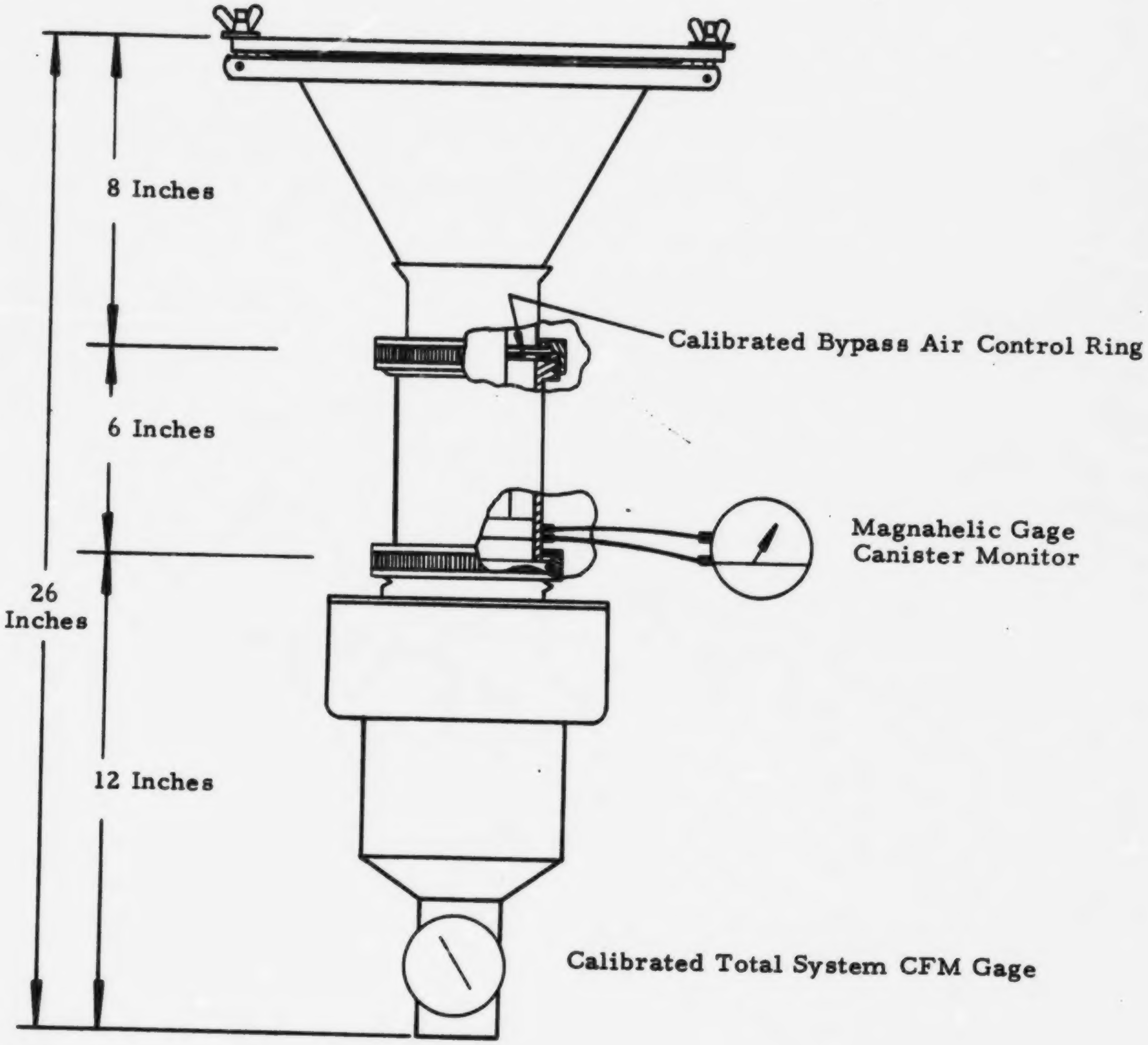

8 Inches
Calibrated Bypass Air Control Ring
6 Inches
Magnahelic Gage
Canister Monitor
26
Inches
12 Inches
Calibrated Total System CFM Gage

Figure 2-17

Hi-Vol Collection Plenum Installed in Place

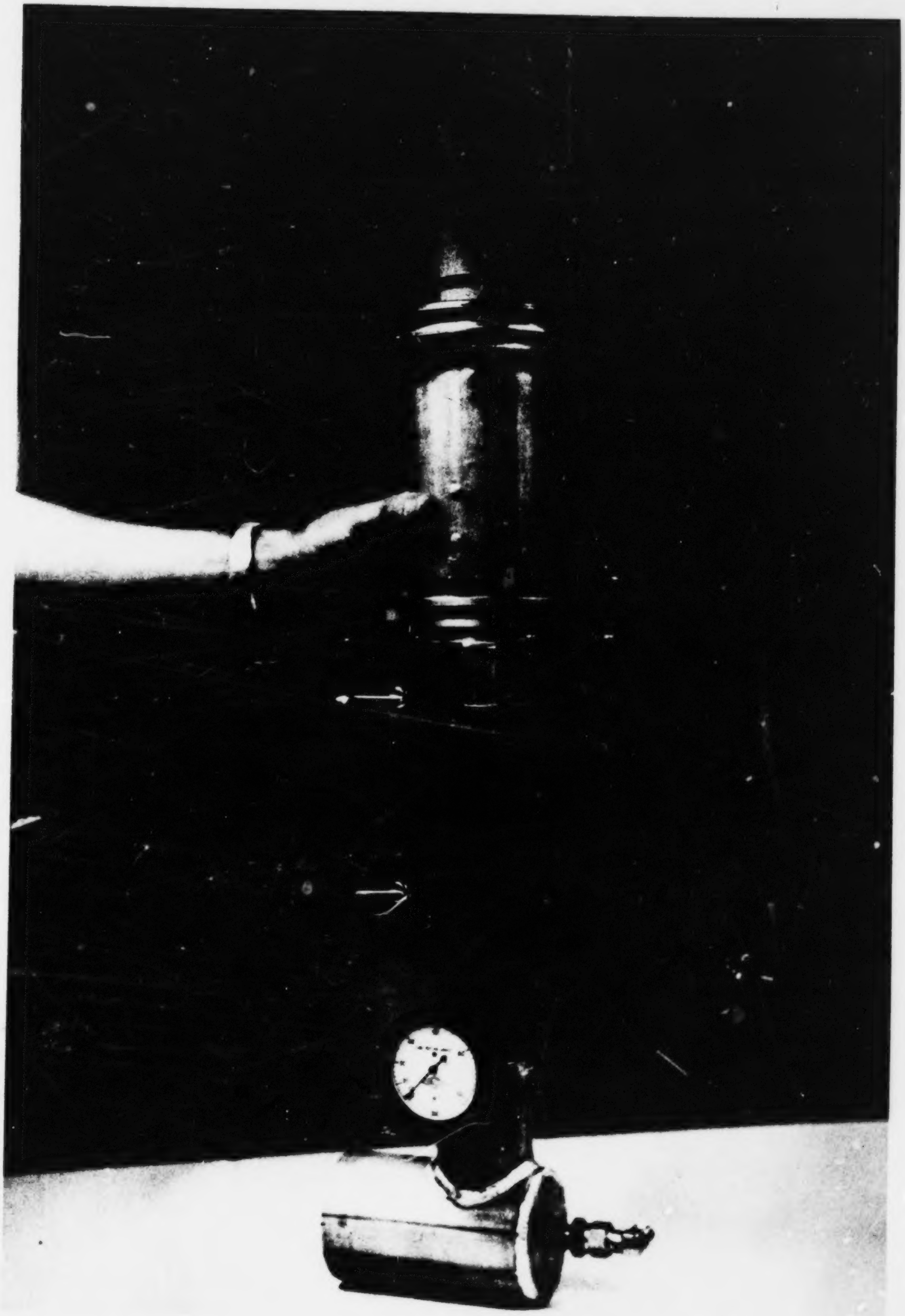


Figure 2-18
Collection Plenum and Canister Assembled onto Hi-Vol Sampler

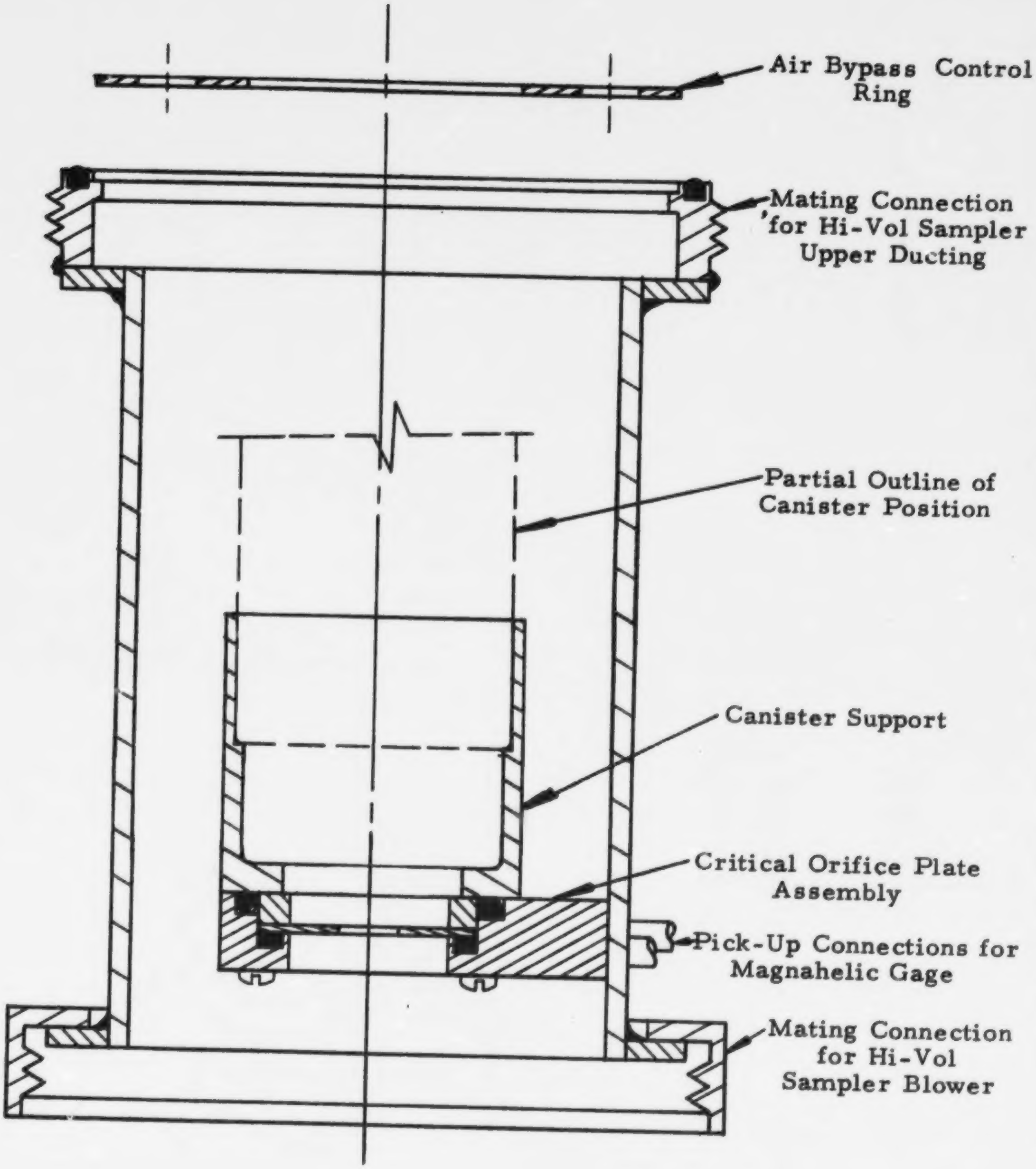

Air Bypass Control Ring
Mating Connection for Hi-Vol Sampler Upper Ducting
Partial Outline of Canister Position
Canister Support
Critical Orifice Plate Assembly
Pick-Up Connections for Magnahelic Gage
Mating Connection for Hi-Vol Sampler Blower

Figure 2-19

Hi-Vol Collection Plenum

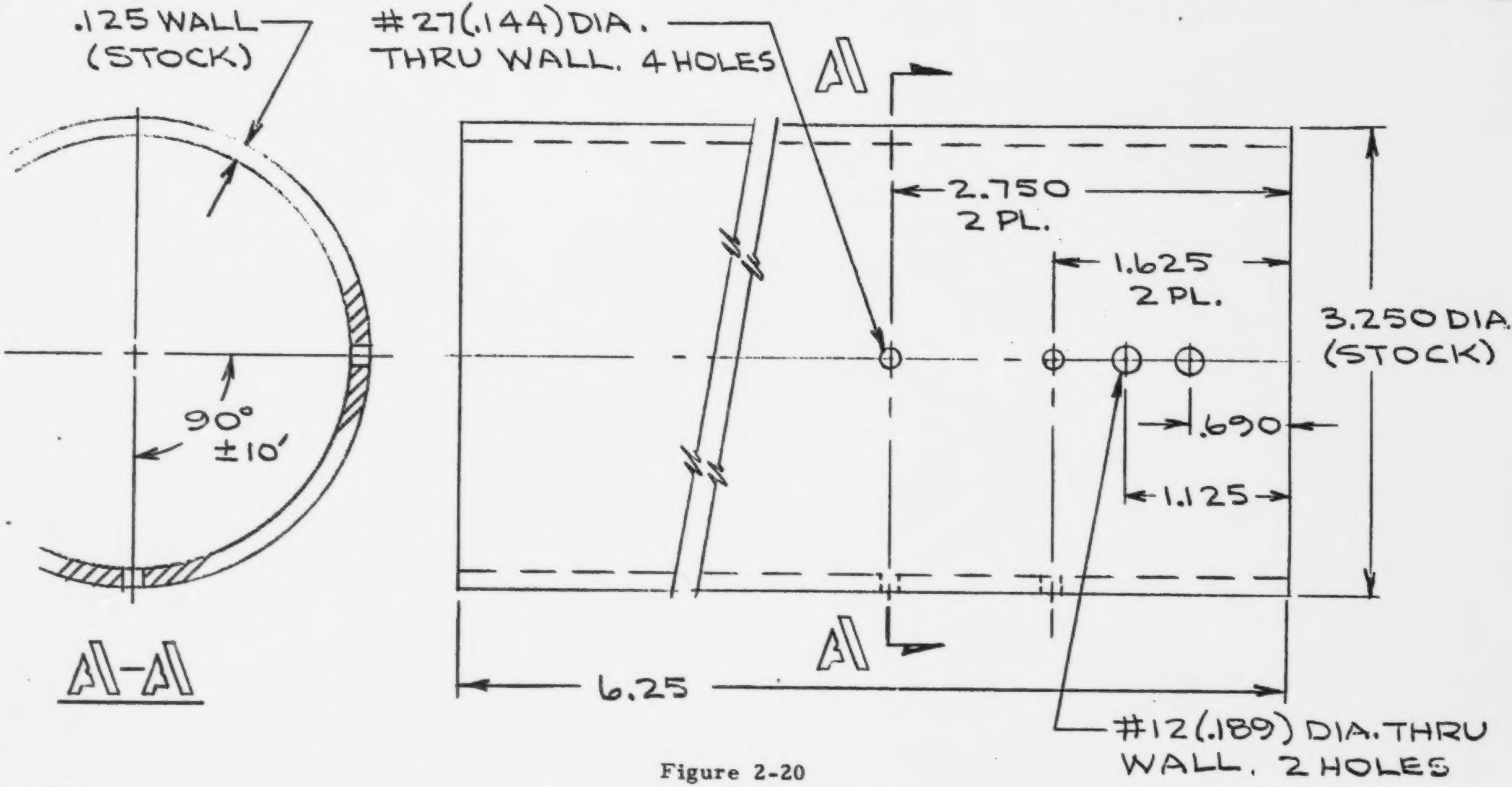

.125 WALL
(STOCK)
90°
±10'
A-A
#27(.144) DIA.
THRU WALL. 4 HOLES
A
A
2.750
2 PL.
1.625
2 PL.
3.250 DIA.
(STOCK)
.690
1.125
6.25
#12(.189) DIA. THRU
WALL. 2 HOLES

Figure 2-20

PLENUM TUBE

MAT.: 6061-T6 ALUM.

NO. REQ.:

GEOMET, INC.

1335

DRAWN BY:
H.H. ANDERSON
10/20/72

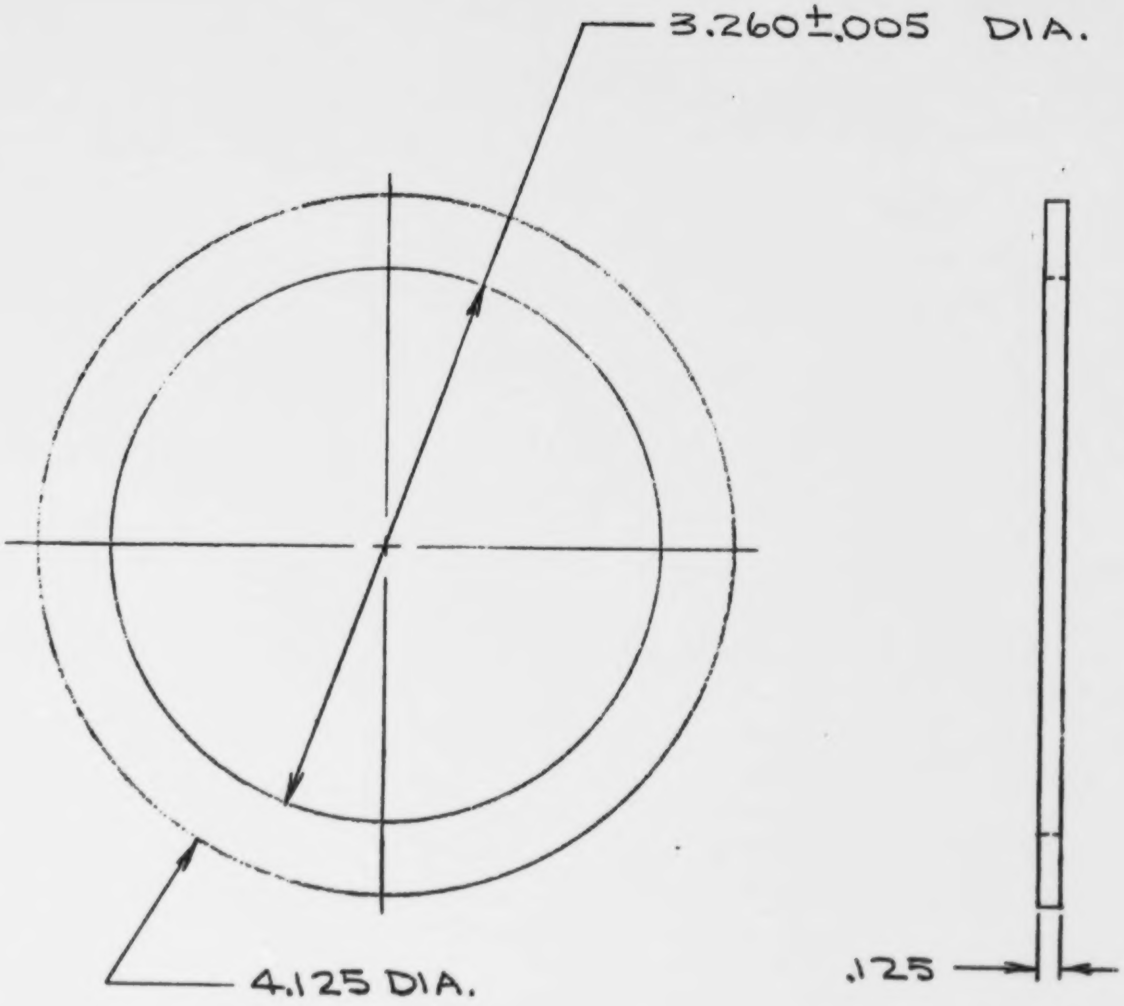

3.260±.005 DIA.
4.125 DIA.
.125

Figure 2-21

FLANGE RING

MAT.: 6061-T6 ALUM.

NO. REQ.:

GEOMET, INC.

1334

DRAWN BY:
H.H. ANDERSON
10/20/72

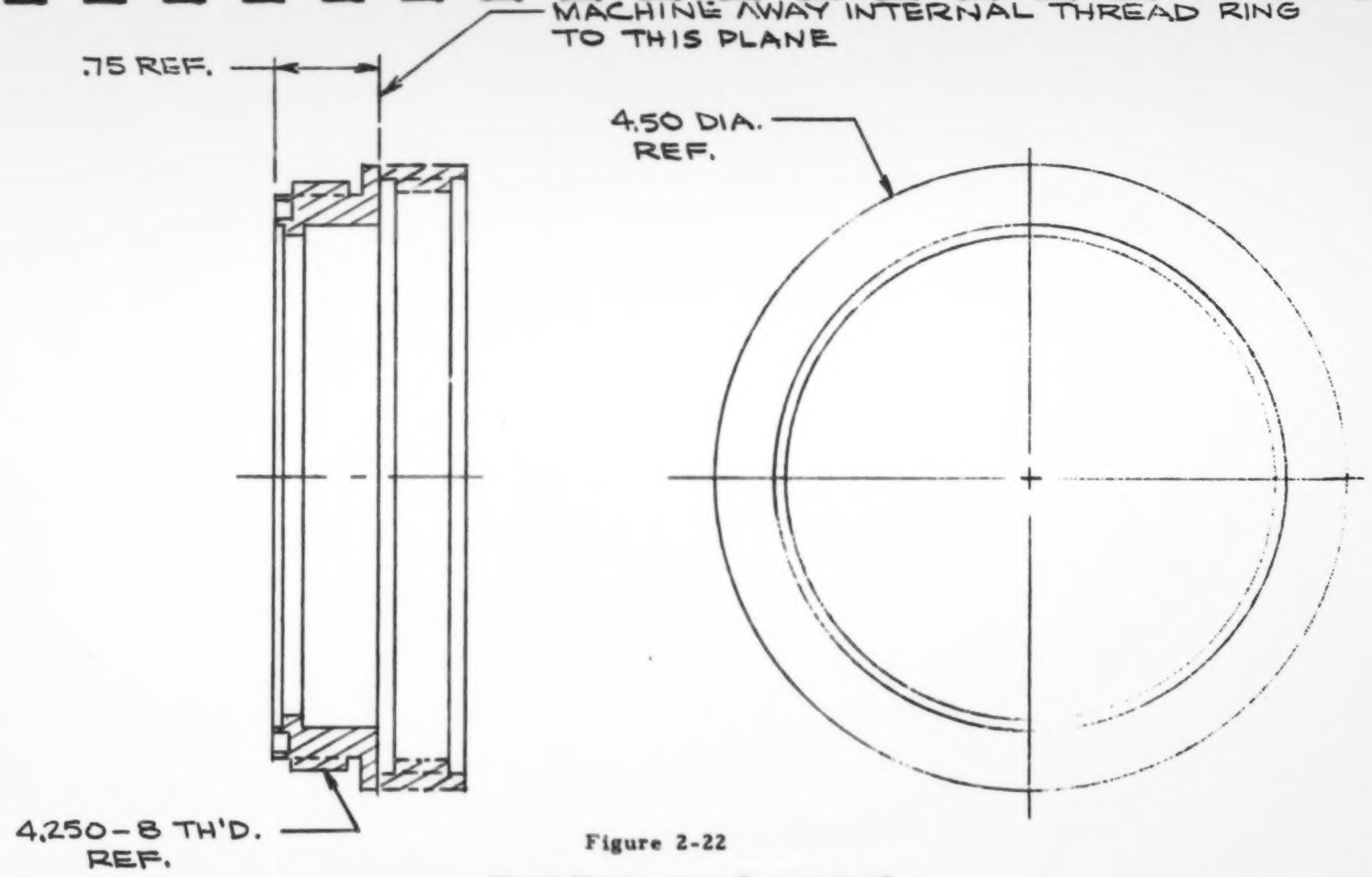

MACHINE AWAY INTERNAL THREAD RING
TO THIS PLANE
.75 REF.
4.50 DIA.
REF.
4.250-8 TH'D.
REF.

Figure 2-22

THREAD RING MODIF.

MAT. :

NO. REQ. :

1336

GEOMET, INC.

DRAWN BY :
H.H. ANDERSON
10/23/72

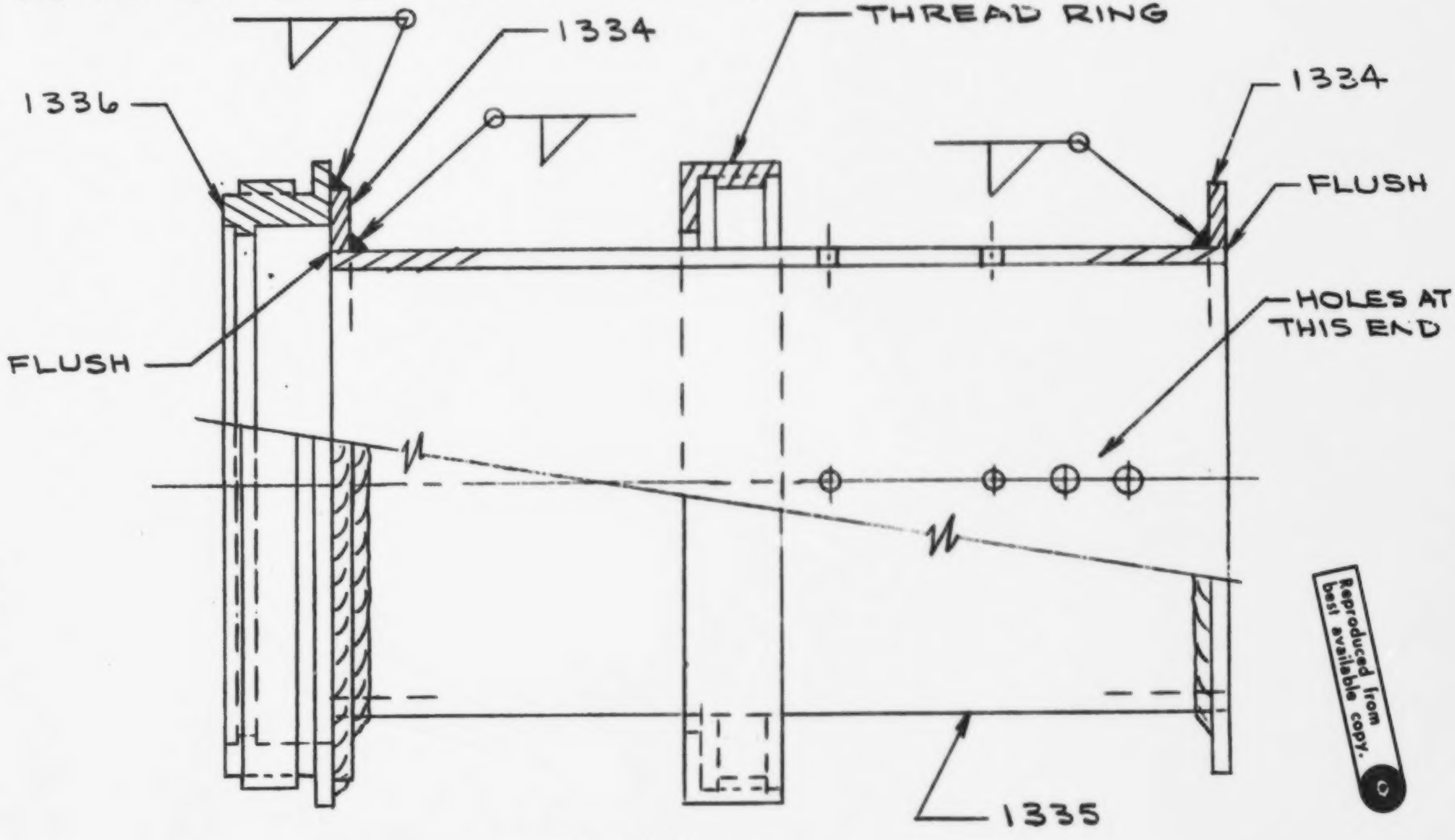

1336
1334
THREAD RING
1334
FLUSH
HOLES AT THIS END
FLUSH
1335

Figure 2-23

PLENUM TUBE WELDMENT

MAT.:
NO REQ.: .

GEOMET, INC.

1337

DRAWN BY:
H.H. ANDERSON
10/23/72

Reproduced from best available copy.

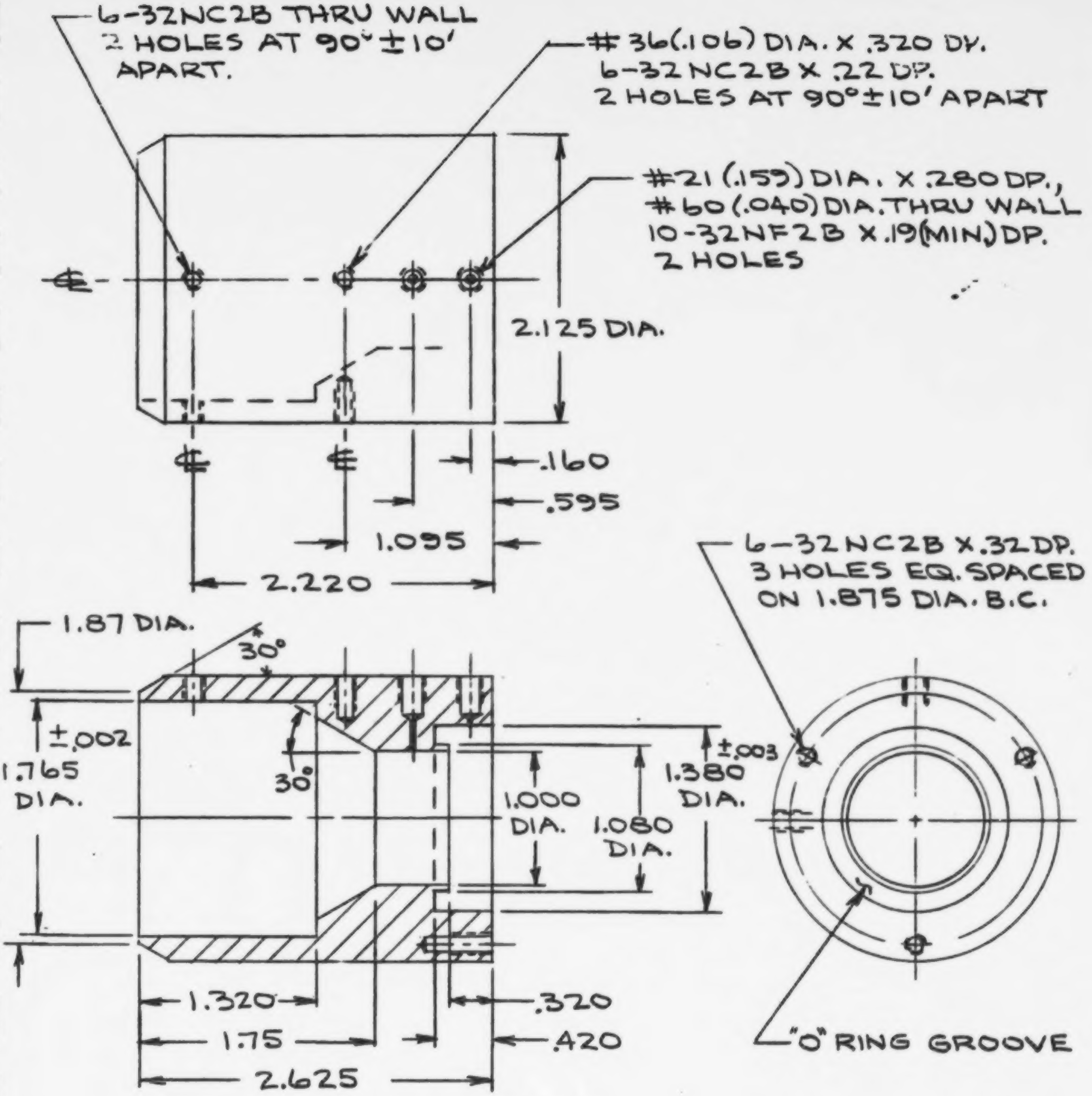

6-32NC2B THRU WALL
2 HOLES AT 90°±10' APART.
#36(.106) DIA. X .320 DP.
6-32NC2B X .22 DP.
2 HOLES AT 90°±10' APART
#21 (.159) DIA. X .280 DP.,
#60 (.040) DIA. THRU WALL
10-32NF2B X .19(MIN.) DP.
2 HOLES
2.125 DIA.
.160
.595
1.095
2.220
6-32NC2B X .32 DP.
3 HOLES EQ. SPACED
ON 1.875 DIA. B.C.
1.87 DIA.
30°
±.002
1.765 DIA.
30°
1.000 DIA.
1.080 DIA.
±.003
1.380 DIA.
1.320
.320
1.75
.420
2.625
"O" RING GROOVE

Figure 2-24

ORIFICE HOUSING

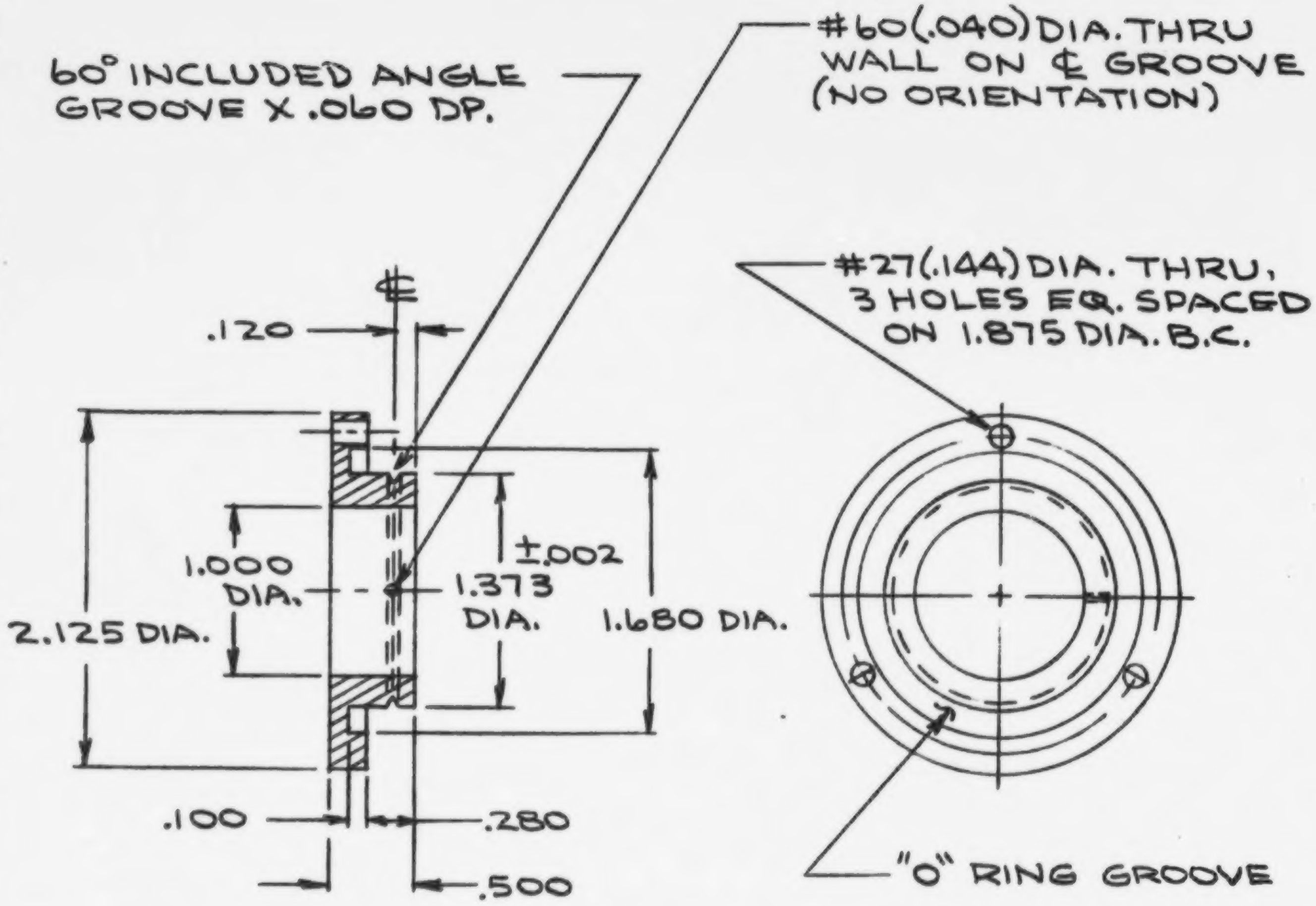
MAT.: 6061-T6 ALUM.

NO. REQ.:

GEOMET, INC.

1338

DRAWN BY:
H.H.ANDERSON
10/23/7



60° INCLUDED ANGLE GROOVE X .060 DP.
#60(.040) DIA. THRU WALL ON ℄ GROOVE (NO ORIENTATION)
℄
.120
1.000 DIA.
±.002
1.373 DIA.
1.680 DIA.
2.125 DIA.
.100
.280
.500
#27(.144) DIA. THRU, 3 HOLES EQ. SPACED ON 1.875 DIA. B.C.
"O" RING GROOVE

Figure 2-25

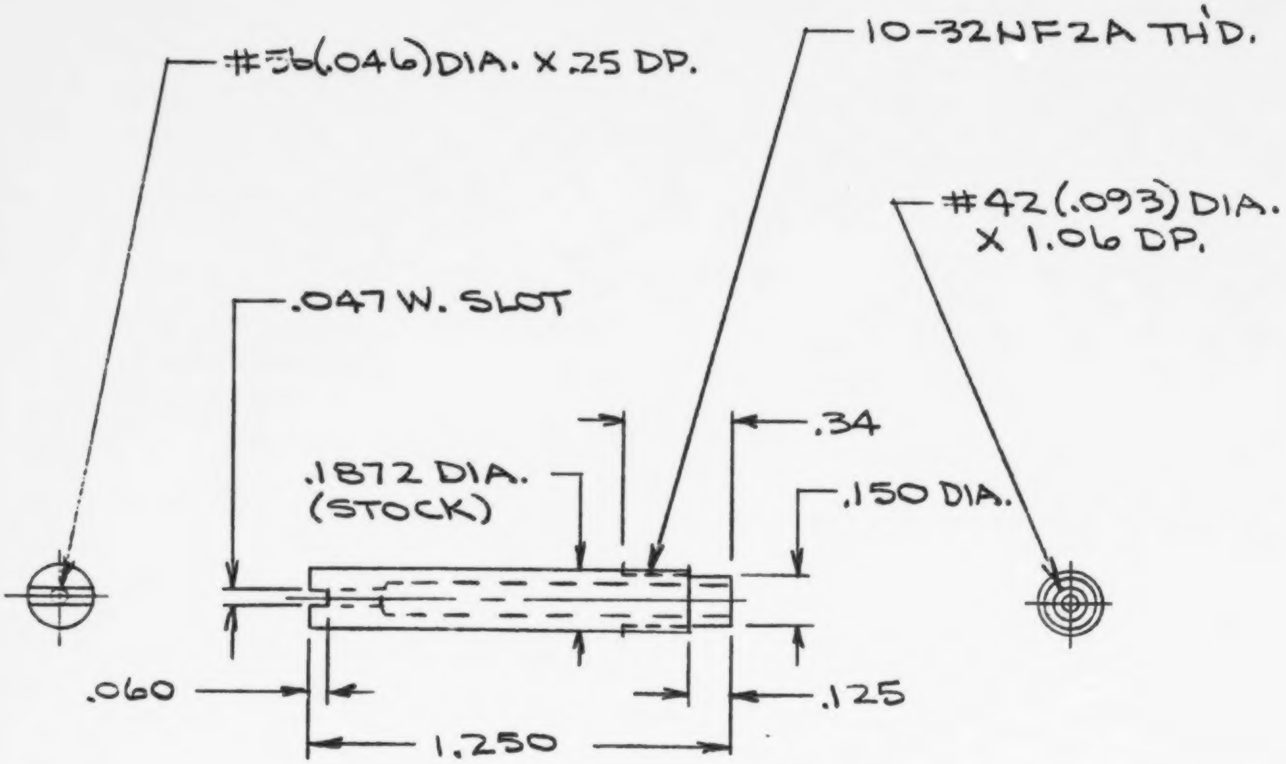
RETAINING RING

MAT.: 6061-T6 ALUM.
NO. REQ.:

GEOMET, INC.

1339

DRAWN BY:
H.H. ANDERSON
10/24/72



#56(.046) DIA. X .25 DP.
10-32NF2A THD.
#42(.093) DIA.
X 1.06 DP.
.047 W. SLOT
.34
.1872 DIA.
(STOCK)
.150 DIA.
.060
.125
1.250

Figure 2-26

PRESSURE TUBE

MAT.: 303 CRES
NO. REQ.:

GEOMET, INC.

1344

DRAWN BY:
H.H. ANDERSON
10/26/72

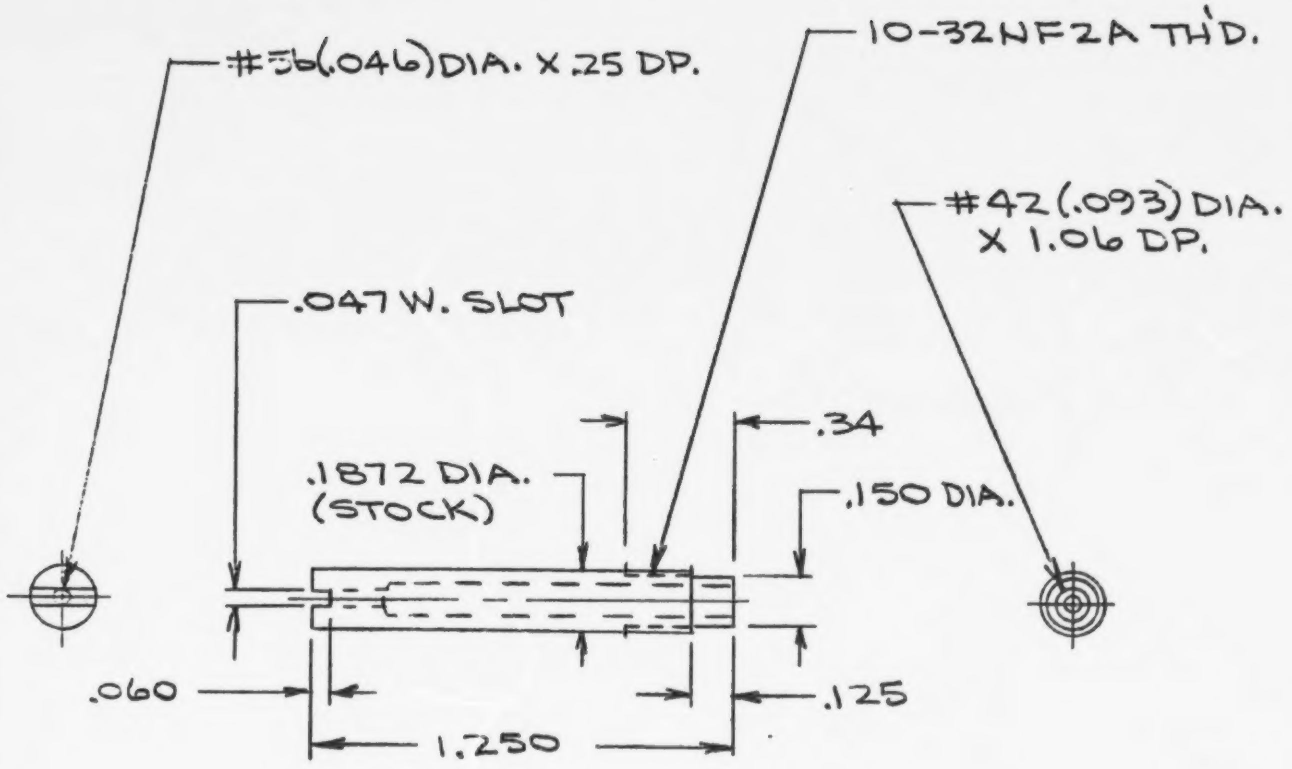

#56(.046) DIA. X .25 DP.
10-32NF2A THD.
#42(.093) DIA. X 1.06 DP.
.047 W. SLOT
.34
.1872 DIA. (STOCK)
.150 DIA.
.060
.125
1.250

Figure 2-26

PRESSURE TUBE

MAT.: 303 CRES
NO. REQ.:

GEOMET, INC.

1344

DRAWN BY:
H.H. ANDERSON
10/26/72

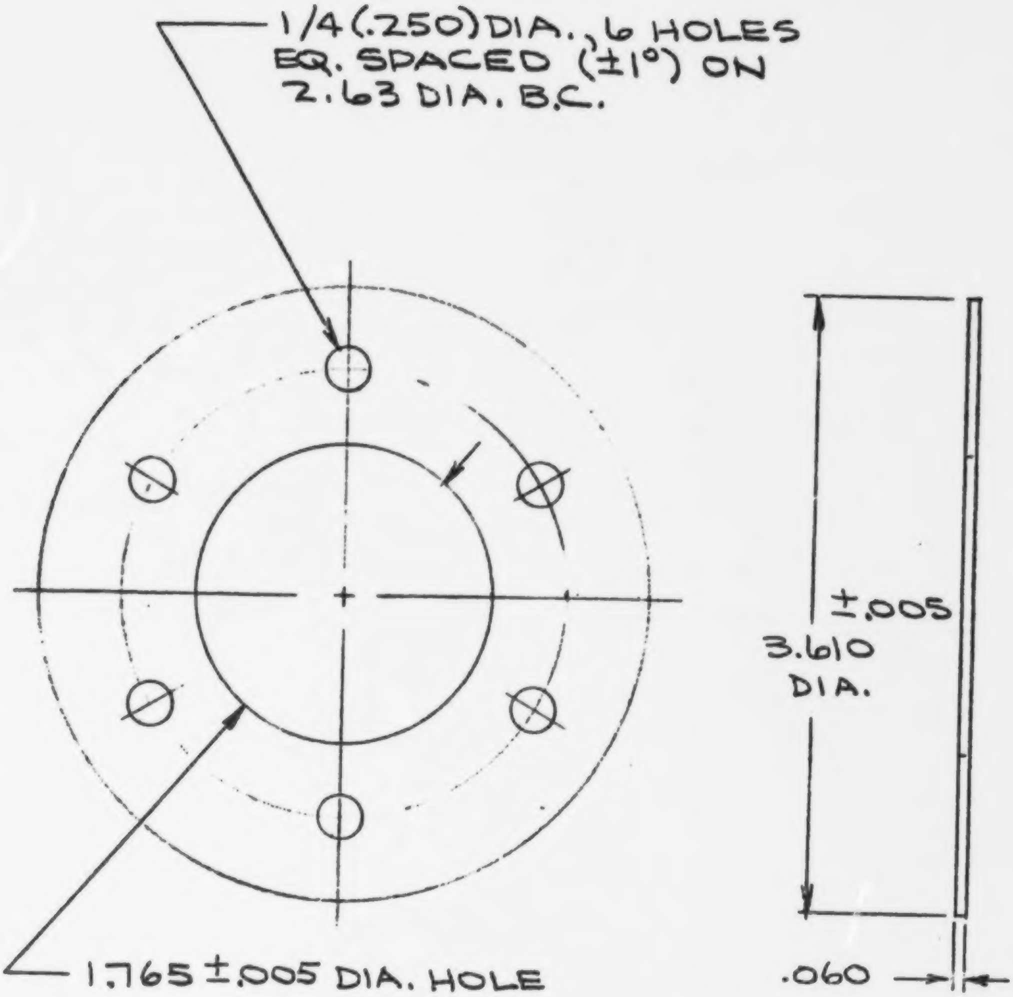

1/4 (.250) DIA., 6 HOLES
EQ. SPACED (±1°) ON
2.63 DIA. B.C.
±.005
3.610
DIA.
1.765 ±.005 DIA. HOLE
.060

Figure 2-27

AIR BYPASS CONTROL RING

MAT.: 6061-T6 ALUM.
NO. REQ.:

GEOMET, INC.

1340

DRAWN BY:
H.H. ANDERSON
10/24/72

2.7.3 Collection Canister Design

The physical characteristics of the Prototype system canisters were established as a result of extensive studies of Hi-Vol air transport characteristics, mercury collection material surface area and packing parameters, and evaluation of the wide range of mercury levels which must be collected and recovered in analysis. The canisters were scaled down from a 1000 gm volume to 180 grams (~160 ml) of adsorbent for elemental mercury collection, and a like volume containing 75 grams of absorbent for organic mercury collection. These canister volumes, at ~ 20% air through-put of the Hi-Vol total at 20 CFM, provide the ability to collect and analyze over the entire collection range from a few nanograms of mercury to 100,000 is ng/M^3 as stipulated in the contract Statement of Work.

The canisters will be utilized for mercury collection, sample transport and storage and are therefore to be light weight and durable. PVC plastic canisters are well suited for this use, and heavier more expensive materials of construction (e.g. stainless steel) were therefore unwarranted. The canister bodies, screen closures and plugs are recyclable, and easily cleaned for reuse. A drawing depicting the assembly of the canisters within the Hi-Vol plenum is provided in Figure 2-28.

The design for the prototype collection canisters is provided in Figures 2-29 through 2-31. The canister bodies are fabricated from sections of plastic tubing and closures which retain the absorbents are held in place by a close-tolerance friction fit. The Type "A" end closures, Figure 2-29, are fabricated with a captive screen section which prevents absorbent loss yet allows air sample through-put. The center dividing closure, Type "B" configuration, is fabricated by joining together parts of two Type "A" closures. This minimizes the number of special fittings and decreases tooling and fabrication costs. The canisters may be easily emptied and refilled in the field, or shipped intact in plastic shipping containers back to a central laboratory for analysis and recycling.

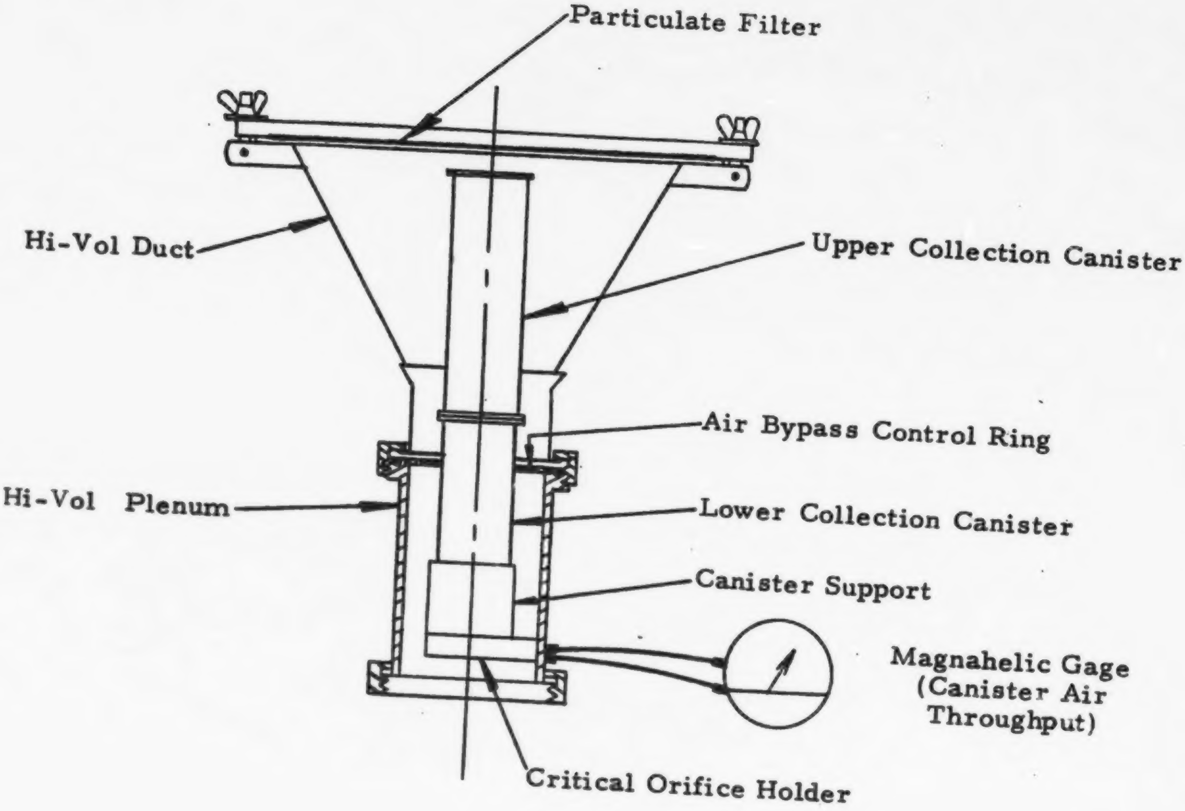

Particulate Filter
Hi-Vol Duct
Upper Collection Canister
Air Bypass Control Ring
Hi-Vol Plenum
Lower Collection Canister
Canister Support
Magnahelic Gage
(Canister Air
Throughput)
Critical Orifice Holder

Figure 2-28

Collection Canister/Hi-Vol Sampler Prototype
Interface Configuration

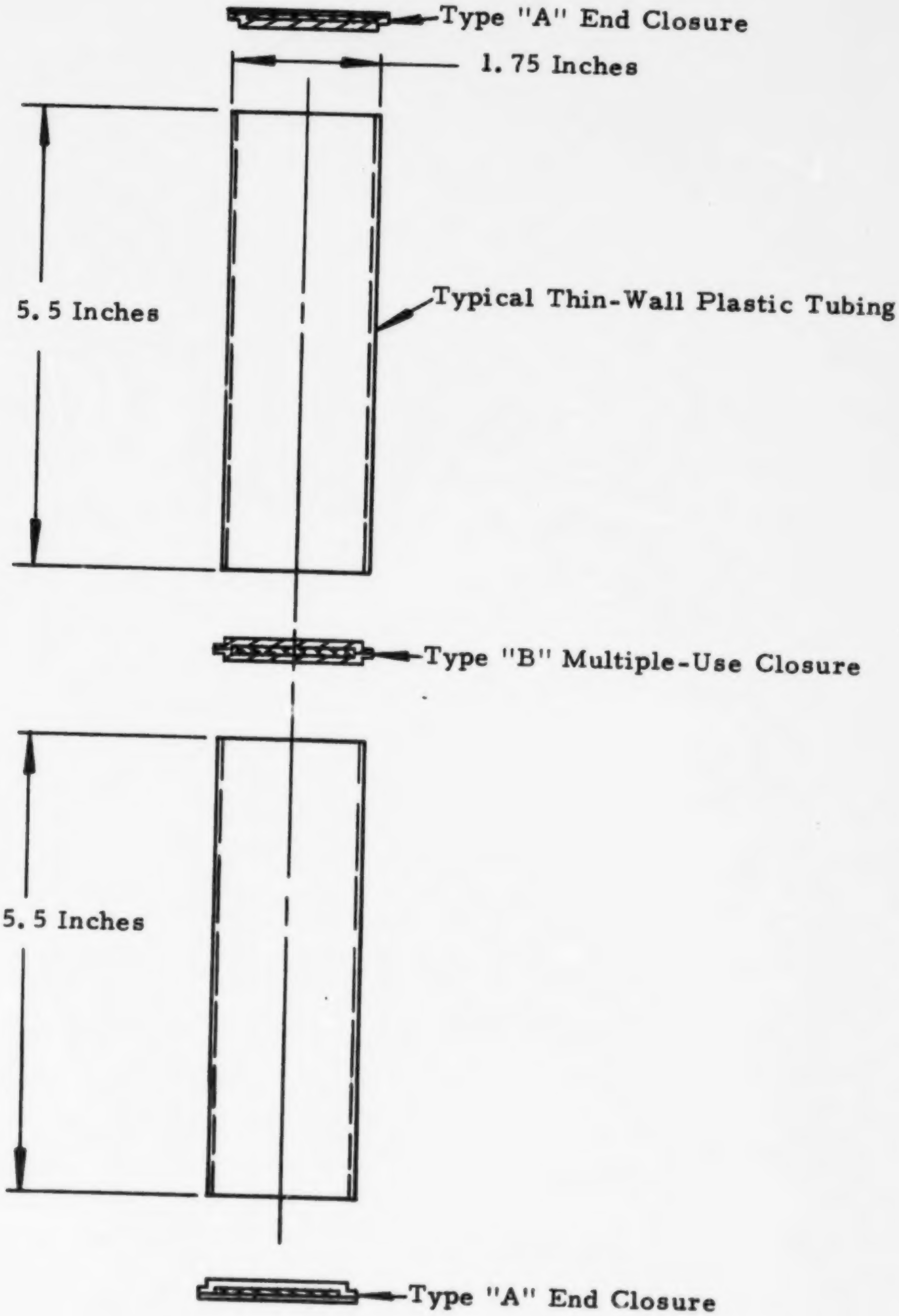

Type "A" End Closure
1.75 Inches
5.5 Inches
Typical Thin-Wall Plastic Tubing
Type "B" Multiple-Use Closure
5.5 Inches
Type "A" End Closure

Figure 2-29

Prototype Multiple-Use Collection Canisters

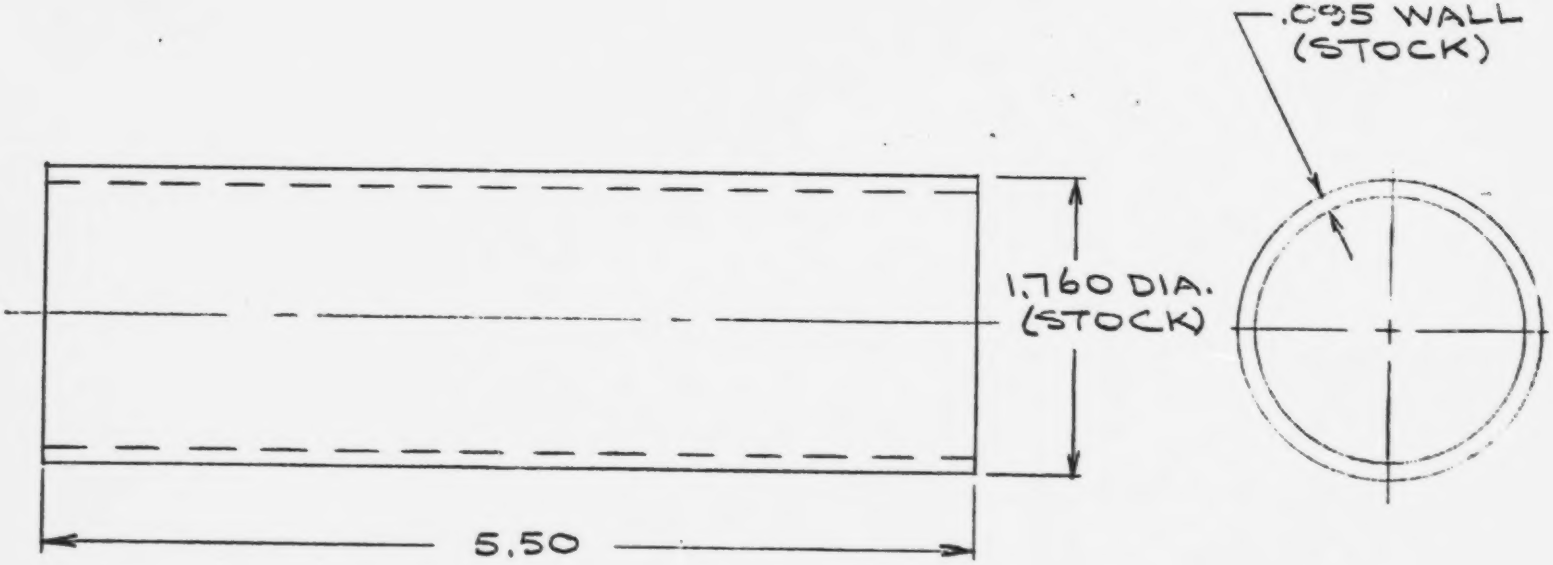

.095 WALL
(STOCK)
1.760 DIA.
(STOCK)
5.50

Figure 2-30

CANISTER BODY

MAT.: E.M.A. VT-175 (ABS-GREY)
NO. REQ.:

GEOMET, INC.

1341

DRAWN BY:
H.H. ANDERSON
10/24/72

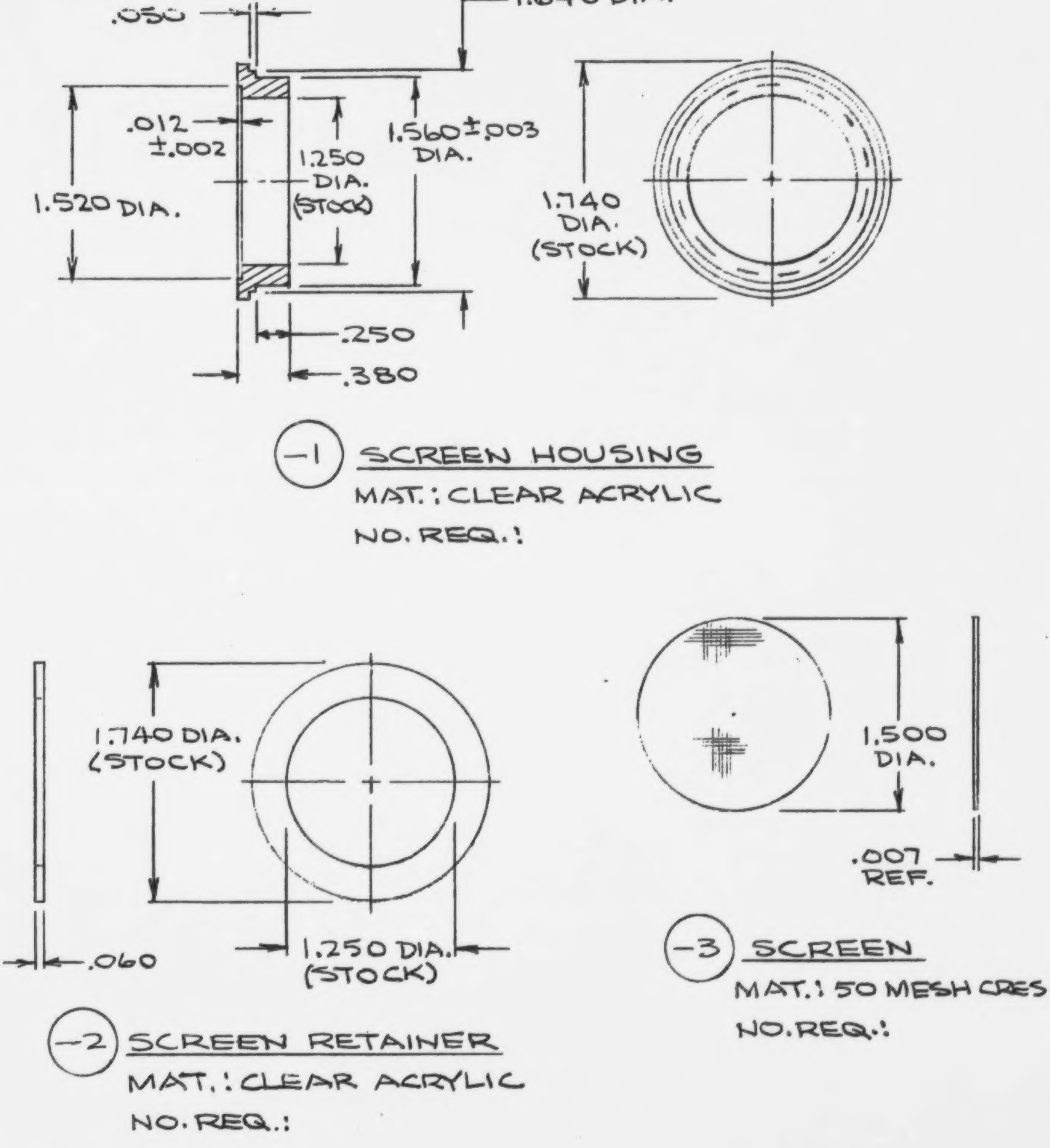

.050
1.640 DIA.
.012
±.002
1.250
DIA.
(STOCK)
1.560±.003
DIA.
1.520 DIA.
1.740
DIA.
(STOCK)
.250
.380
-1 SCREEN HOUSING
MAT.: CLEAR ACRYLIC
NO. REQ.:
1.740 DIA.
(STOCK)
.060
1.250 DIA.
(STOCK)
-2 SCREEN RETAINER
MAT.: CLEAR ACRYLIC
NO. REQ.:
1.500
DIA.
.007
REF.
-3 SCREEN
MAT.: 50 MESH CRES
NO. REQ.:

Figure 2-31

GEOMET, INC.

1349

DRAWN BY:
H.H. ANDERSON

2.7.4 Recovery Analysis System

The processing method is fully described in Section 2.8, and with the exception of the recovery furnace is non-deliverable under the contract. The recovery furnace consists of (1) a furnace insert, which is used for holding absorbents within the furnace, and (2) a heavy duty crucible furnace capable of heating samples in the range of 0-2000°F.

The aliquots of absorbents are placed in a modified crucible and attached to a support fixture which is lowered into the crucible furnace. The support fixture has been designed to direct a stream of transport air through the heated collection material and carry off the liberated mercury to a series of EPA-type liquid collection bubblers containing iodine monochloride solution. A detailed assembly drawing and fabrication sketches are provided in Figures 2-32 through 2-38.

2.7.5 Demonstration to EPA Program Monitor

Phase V, of the contract Statement of Work, required that GEOMET conduct a demonstration of the operation of the Prototype collection device before the cognizant Project Officer at the National Environmental Research Center, Research Triangle Park, North Carolina.

In light of the attendant processing equipment, recalibration procedures and nature of the collection device, the cognizant Project Officer was invited to witness a demonstration of the Prototype instrumentation at the laboratory of the GEOMET Office of Experimental Development in Pomona, California. On Tuesday, 20 February, 1973, Miss Eva Wittgenstein, Program Project Officer (EPA Laboratory Measurements Research Section, Chemistry and Physics Laboratory), reviewed the development effort on the program and witnessed an operational demonstration of the Prototype instrumentation.

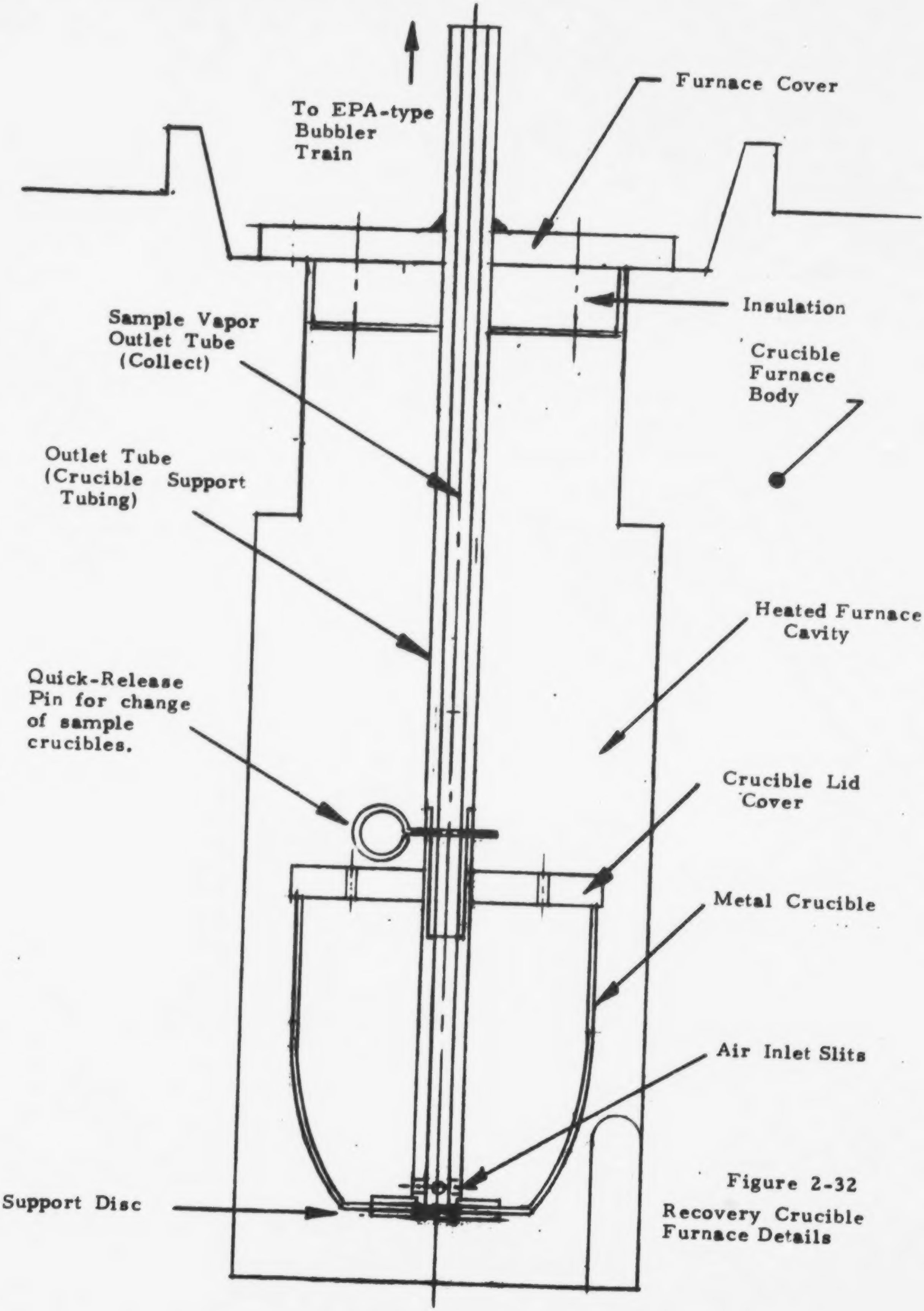
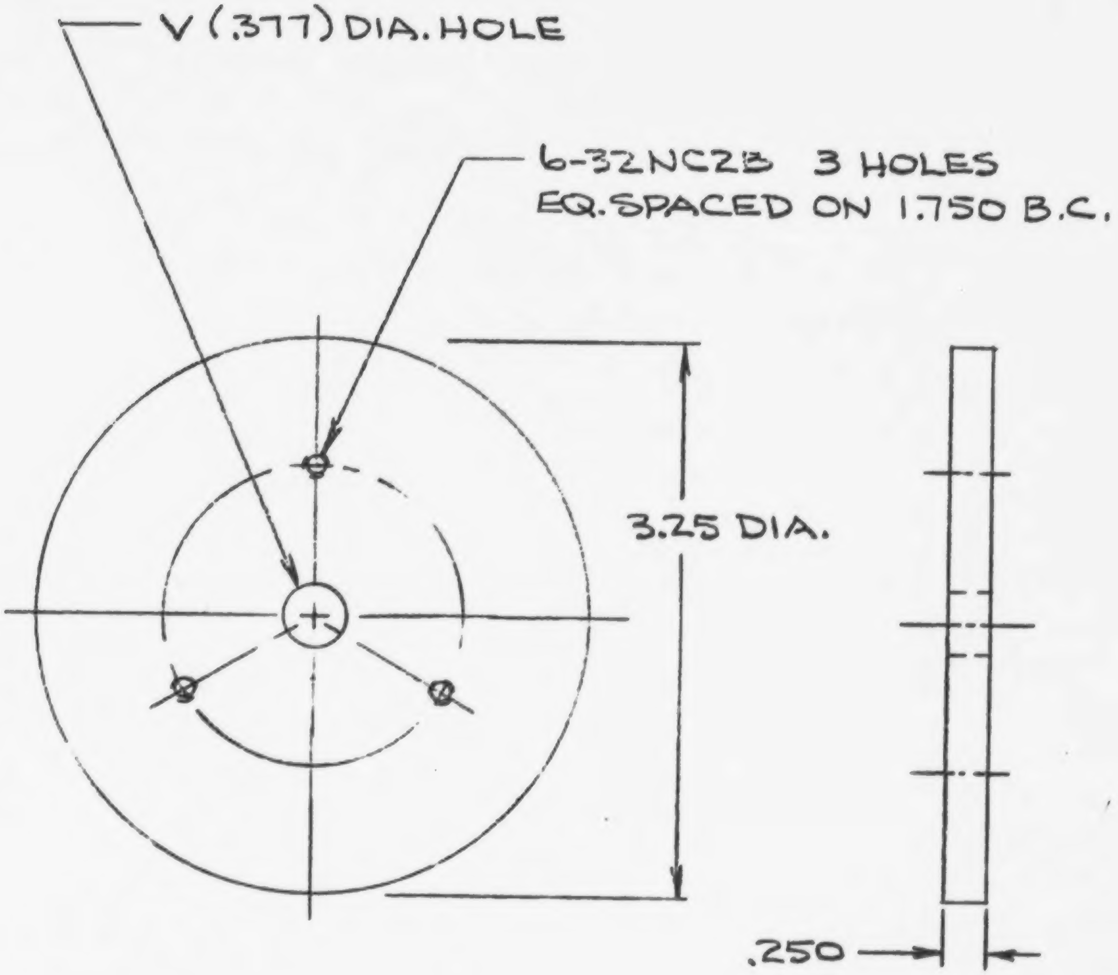

To EPA-type Bubbler Train
Furnace Cover
Insulation
Sample Vapor Outlet Tube (Collect)
Crucible Furnace Body
Outlet Tube (Crucible Support Tubing)
Heated Furnace Cavity
Quick-Release Pin for change of sample crucibles.
Crucible Lid Cover
Metal Crucible
Air Inlet Slits
Support Disc

Figure 2-32
Recovery Crucible Furnace Details


V (.377) DIA. HOLE
6-32NC2B 3 HOLES
EQ. SPACED ON 1.750 B.C.
3.25 DIA.
.250

FURNACE COVER

MAT.: 302-304 CRES

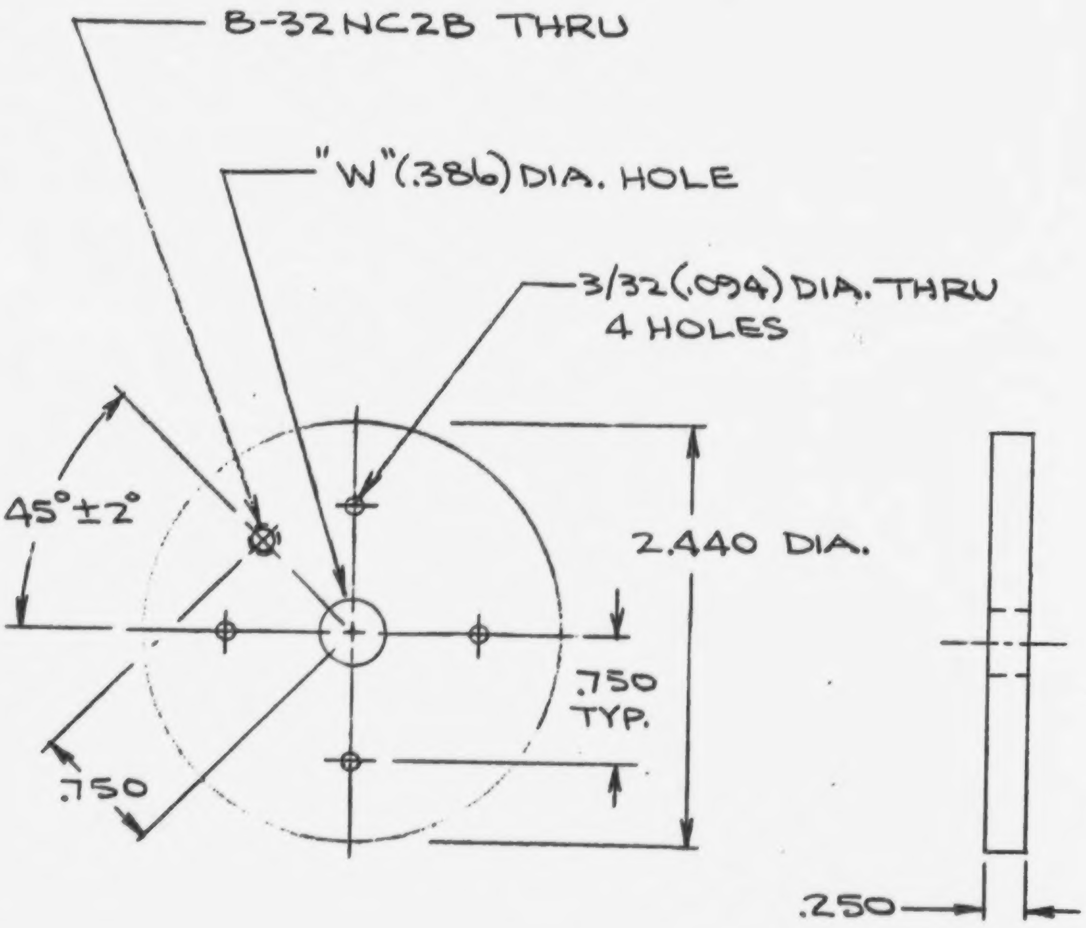
NO. REQ.:

Figure 2-33

GEOMET, INC.

1370

DRAWN BY:
H.H. ANDERSON


8-32NC2B THRU
"W"(.386) DIA. HOLE
3/32(.094) DIA. THRU
4 HOLES
45°±2°
2.440 DIA.
.750
TYP.
.750
.250

CRUCIBLE COVER

MAT.: 302-304 CRES

NO. REQ.:

Figure 2-34

GEOMET, INC.

1371

DRAWN BY:
H.H.ANDERSON

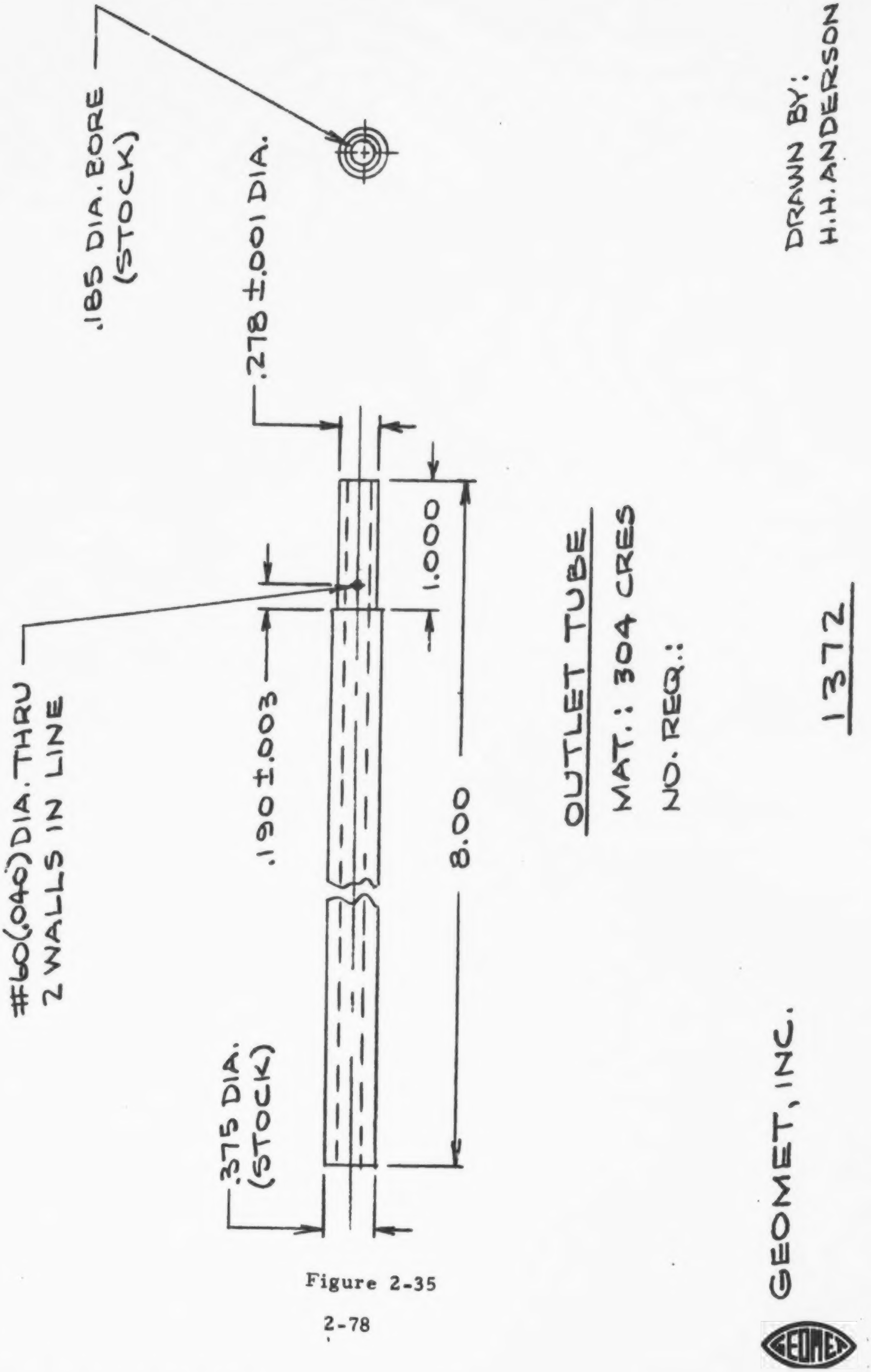
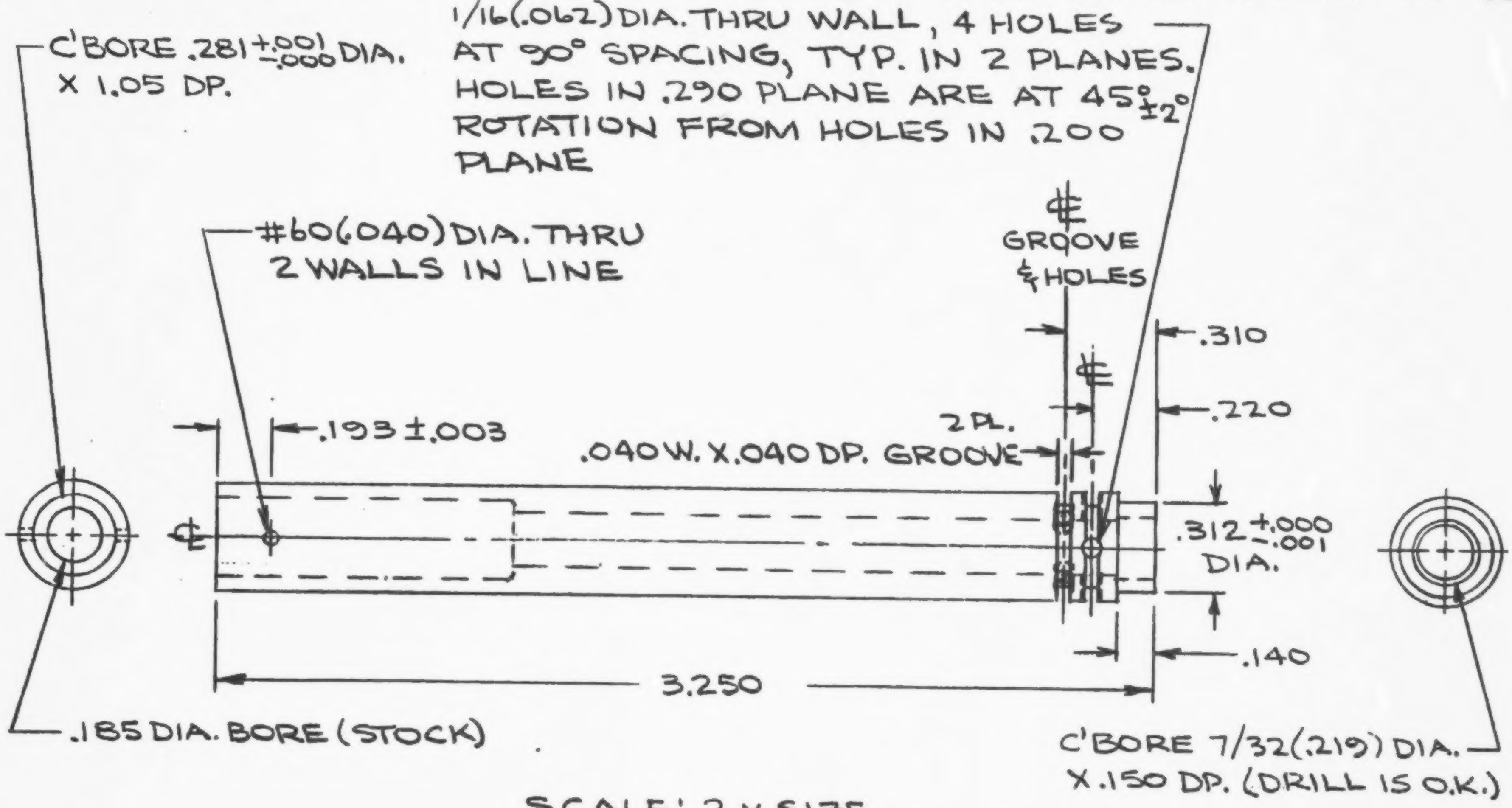

.185 DIA. BORE
(STOCK)
.278 ±.001 DIA.
1.000
#60(.040) DIA. THRU
2 WALLS IN LINE
.190 ±.003
8.00
.375 DIA.
(STOCK)
OUTLET TUBE
MAT.: 304 CRES
NO. REQ.:
1372
DRAWN BY:
H.H. ANDERSON
GEOMET, INC.

Figure 2-35


C'BORE .281 +.001 -.000 DIA. X 1.05 DP.
1/16(.062) DIA. THRU WALL, 4 HOLES AT 90° SPACING, TYP. IN 2 PLANES. HOLES IN .290 PLANE ARE AT 45° ±2° ROTATION FROM HOLES IN .200 PLANE
#60(.040) DIA. THRU 2 WALLS IN LINE
℄ GROOVE & HOLES
.310
℄
.220
2 PL.
.193 ±.003
.040 W. X .040 DP. GROOVE
℄
.312 +.000 -.001 DIA.
.140
3.250
.185 DIA. BORE (STOCK)
C'BORE 7/32(.219) DIA. X .150 DP. (DRILL IS O.K.)

SCALE: 2 X SIZE

COLLECT TUBE

MAT.: 304 CRES

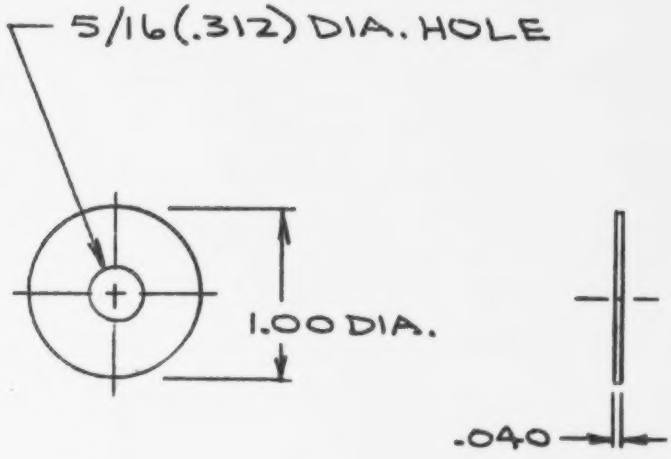
NO. REQ.:

Figure 2-36

GEOMET, INC.

1373

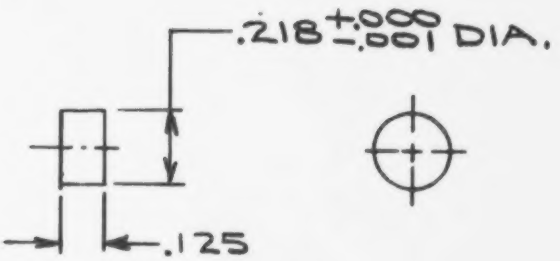
DRAWN BY:
H.H. ANDERSON


5/16(.312) DIA. HOLE
1.00 DIA.
.040

(-1) WASHER

MAT.: 302-304 CRES

NO. REQ.:


.218 $^{+.000}_{-.001}$ DIA.
.125

SCALE: 2X SIZE

(-2) PLUG

MAT.: 302-304 CRES

NO. REQ.:

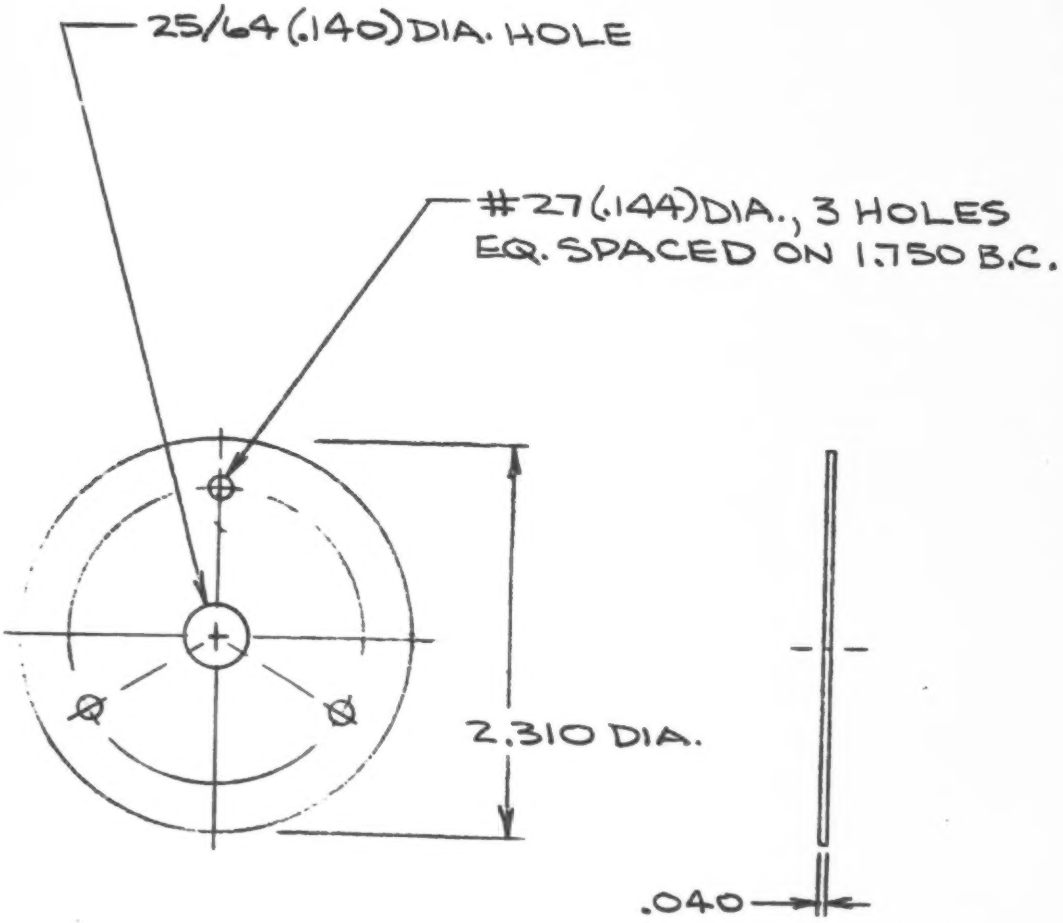
CRUCIBLE DETAILS

Figure 2-37

GEOMET, INC.

DRAWN BY:
H.H. ANDERSON

1374


25/64 (.140) DIA. HOLE
#27 (.144) DIA., 3 HOLES
EQ. SPACED ON 1.750 B.C.
2.310 DIA.
.040

DISC

MAT.: 302-304 CRES

NO. REQ.:

Figure 2-38

GEOMET, INC.

DRAWN BY:
H.H. ANDERSON

1375

2.8 RECOVERY ANALYSIS PROCEDURES

GEOMET was charged with the responsibility of providing both a "removal" and "transfer" step as necessary in quantitative recovery and analysis of all three forms of mercury. The following sections describe the development of this methodology and attendant equipment.

2.8.1 Recovery System Description

The recovery system for the Prototype mercury collection instrumentation was initially the same for all three forms of collected mercury. It was based on a furnace and bubbler technique. As the program progressed, it became apparent that the elemental mercury adsorbent and organic mercury absorbent could be handled by this method, but a better procedure was developed for particulate mercury recovery from the glass fiber filter.

The final system, referring first to the adsorbent (silver-treated alumina for elemental mercury vapor) and the absorbent (activated charcoal for organic mercury forms) is shown in Figures 2-39 and 2-40. The mercury sources feed elemental and organic mercury forms to the Hi-Vol sampler. A portion of the total air stream is metered through the canisters where the two forms of mercury are selectively collected. Following the collection cycle, the two canisters are emptied into storage containers, labeled and sealed. A ten (10) percent aliquot of the adsorbent is weighed out for processing. An equal amount of the original adsorbent, which was not used in the collection cycle, is placed into the resistance

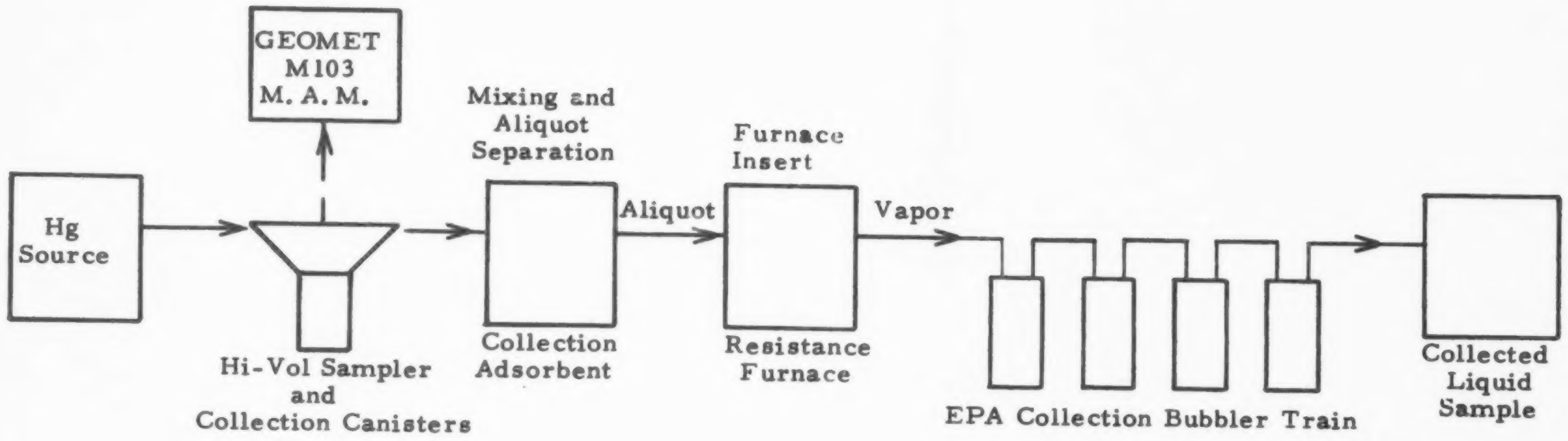

GEOMET
M103
M. A. M.
Hg
Source
Hi-Vol Sampler
and
Collection Canisters
Mixing and
Aliquot
Separation
Collection
Adsorbent
Aliquot
Furnace
Insert
Resistance
Furnace
Vapor
EPA Collection Bubbler Train
Collected
Liquid
Sample

Figure 2-39 Mercury Challenge Collection Method

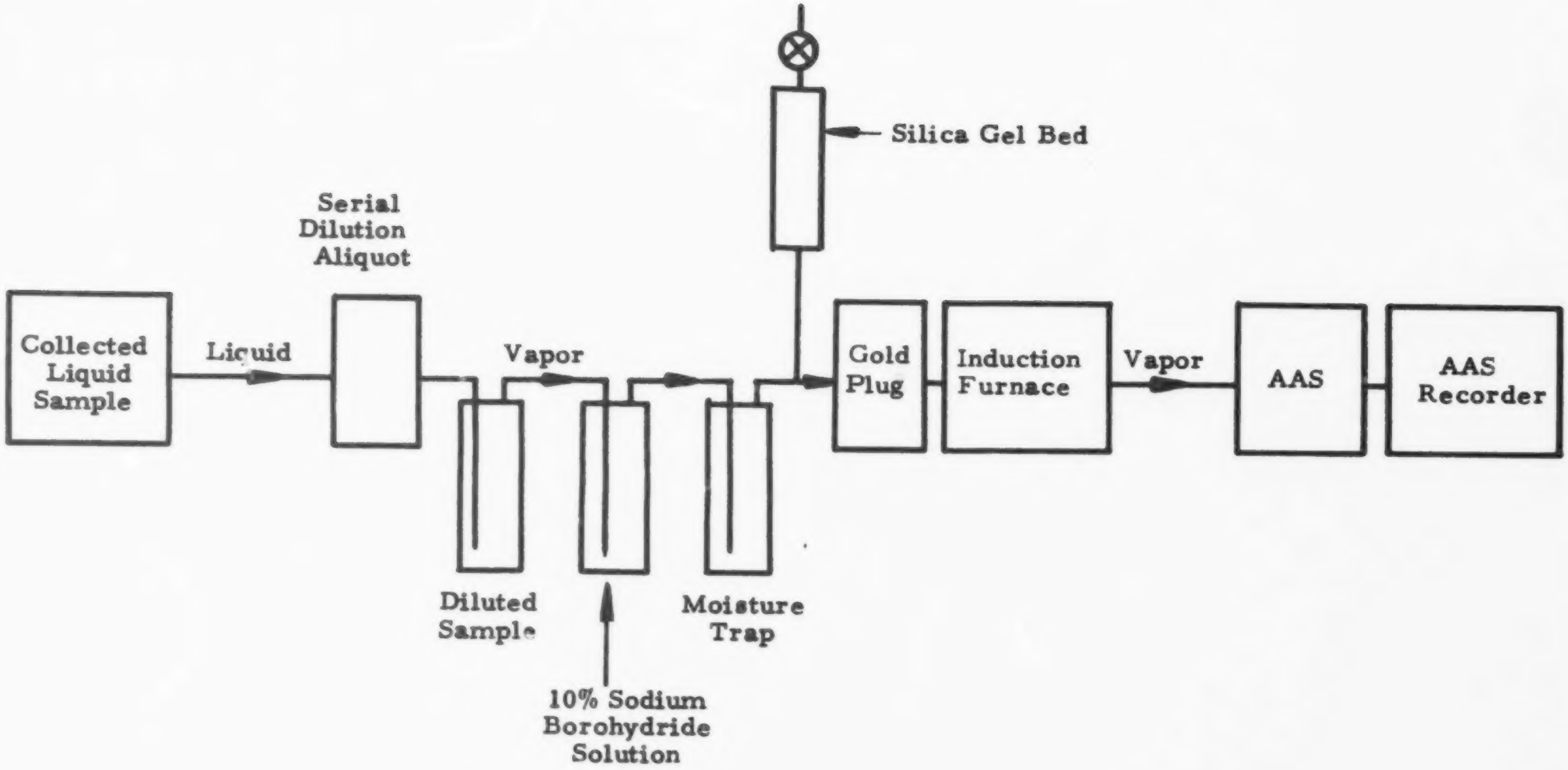

Silica Gel Bed
Serial Dilution Aliquot
Collected Liquid Sample
Liquid
Vapor
Gold Plug
Induction Furnace
Vapor
AAS
AAS Recorder
Diluted Sample
Moisture Trap
10% Sodium Borohydride Solution

Figure 2-40. Analytical Method

A method was devised to fold the filters, on removal from the Hi-Vol sampler following collection, in order to maintain sample integrity and avoid any sample loss. This portion of the processing technique is still valid. There did appear, however, to be some difficulty in obtaining reproducible air flow through the folded filters when they were placed in the recovery furnace for pyrolysis. Therefore, a better and simpler method of analysis processing was developed and tested. The glass filters were folded into a compact bundle (~1" square x 1/2" thick) and placed into a wide-mouthed inert plastic jar containing 100 ml of ICl (iodine monochloride) solution. Several 3mm glass beads were added and the jar was tightly capped. The container was then vigorously shaken so that the beads maserated the filter. The mixture was allowed to stand overnight to insure adequate mercury takeup by the ICl solution. Aliquots of the mixture were then centrifuged to separate the filter metrix from the ICl solution. Portions of the ICl were serially diluted as necessary and analyzed through the AAS for recovered mercury. This procedure could be simplified by use of automated shaking devices, or sonic separators in future development of the total Prototype system. However, for the glass fiber filters it works adequately.

2.8.3 Analysis of Adsorbent Pellets

Initial mercury recovery runs, utilizing silver-treated alumina adsorbents for elemental mercury, were made by placing the pellets in an open cylinder positioned in the resistance furnace. Transport air was pulled from the furnace to the EPA-type, iodine-monochloride collection bubblers through stainless steel tubing. These preliminary tests indicate that >90% of the mercury was released when the furnace reached a temperature of ~350°C. This release was in the form of a rather large "pulse" and tail-off of mercury vapor was still in evidence when the furnace reached 505°C. This phenomenon was due in part to thermal gradients found in heating the mercury-laden alumina adsorbent, and to the construction details of the furnace tube. A cast iron tube was then used as a replacement for the alloy steel liner. This alteration, while negating any apparent problems with mercury vapor hold-up during the absorbent heating procedures, still lacked the necessary handling convenience necessary for rapid laboratory analyses. A conceptual design for the final Prototype system was reported to EPA, and that design is provide in Figure 2-41. This exact design was never fully completed. Instead, a less complicated design was formulated (Reference Section 2.7.4 above) and utilized on the remainder of the program. This furnace insert method allowed the use of a standard tube furnace and obviated the need for a cast iron or other metallic furnace cover liner. The need for a human engineered handling system was very important to the success of the adsorbent recovery procedure.

The analytical procedure for recovery of mercury from the absorbent pellets began by removal of the pellets from the collection canister. The pellets should be transferred to plastic shipping/ storage containers, sealed and labeled. At time of analysis, the adsorbent should be well mixed to achieve uniform mixing of the

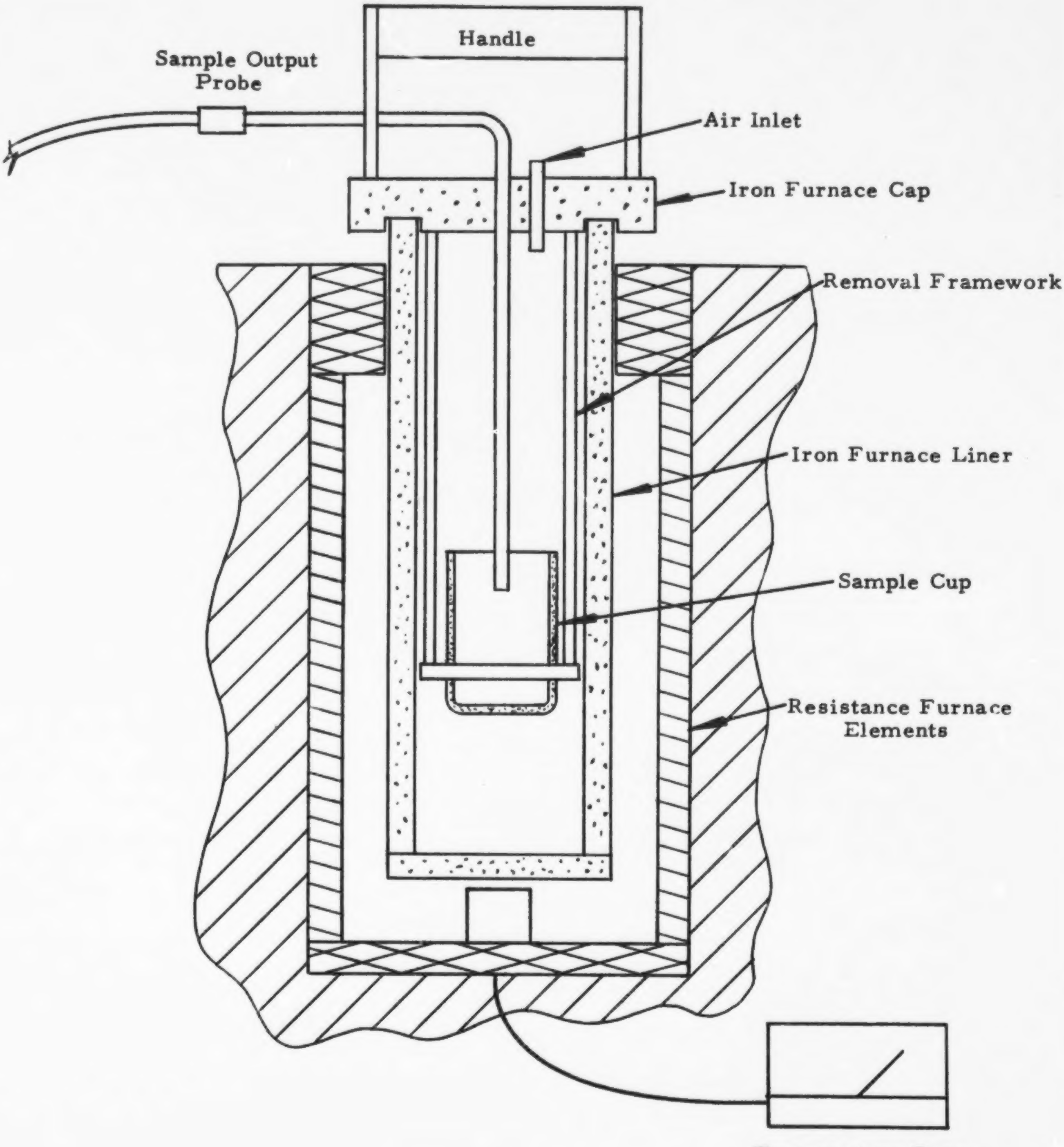

Handle
Sample Output Probe
Air Inlet
Iron Furnace Cap
Removal Framework
Iron Furnace Liner
Sample Cup
Resistance Furnace Elements
Temperature Gage

Figure 2-41

Conceptual Mercury Recovery Resistance Furnace mercury load throughout the adsorbent lot. The final Prototype system canister design holds 180 grams of the silver-treated alumina. A 10% aliquot is weighed out and placed into a cold crucible, assembled onto the furnace insert, and placed within the pre-heated resistance furnace ($\sim$600-700°C) and connected to the EPA collection bubbler train. Air is drawn through the pellet bed and into the bubbler tain for 30 minutes. This has proven to be sufficient time to heat the adsorbent and transport all the elemental mercury into the liquid (ICl) collection bubblers. The 45 ml bubbler volume is collected in clear glass storage bottles and transfered to the processing apparatus described in Section 2.8.1, above.

Refinements were made over the duration of analytical runs made during this program. The tubing from the furnace to the EPA-type collection bubbler train was reduced to a minimum to prevent any fallout of mercury being transported to the bubblers.

Extremely high AAS readouts were occasionally experienced when firing the collected mercury (from the samples obtained out of the recovery process furnace bubblers) off the noble metal wire plug in the induction furnace. In one or two instances, this phenomenon occurred during the recheck of bubbler and pellet blanks. In order to circumvent the possibility of elemental mercury vapor induced by external or other laboratory procedures from interfering with ongoing AAS readouts of critical test runs, two additional bubblers were installed in the vapor transport tubing. The first of these contains approximately 10 ml of a $\geq$10% sodium borohydride solution and is located immediately before the sample bubblers containing the alkaline hydroxylamine solution used in this modified Hatch and Ott procedure. The second is a spray trap. It was found that apparently some portions of the reducing solution used in the analytical method pass into the tubing to the induction furnace from the bubblers. The air stream transports droplets which line the passages and subsequently trap elemental mercury vapor. This mercury source apparently causes intermittent, inexplicable signals in the AAS. The additional sodium borohydride bubbler scrubs off any interferences and droplets, but allows the passage of mercury vapor to the AAS. The second bubbler is used completely empty. It acts as a spray trap and as additional protection for moisture removal from the sample air stream. The basic sample recovery and analytical method utilizing the resistance furnace, modified Hatch and Ott procedure, modified analytical method, induction furnace and AAS was fully successful in processing the silver-treated alumina mercury adsorbent, and the activated charcoal mercury absorbent.

2.8.4 Analysis of Charcoal Absorbent

The activated charcoal used to absorb inorganic and organometallic mercury vapors was analyzed in the same manner as the adsorbent pellets. This method was both practical, from a technical point of view, and economical. It allowed the same equipment developed for the processing and analysis of the pellets to be used for the activated charcoal. The only minor difference in the procedure was that the total charge per canister of charcoal was 75 grams rather located immediately before the sample bubblers containing the alkaline hydroxylamine solution used in this modified Hatch and Ott procedure. The second is a spray trap. It was found that apparently some portions of the reducing solution used in the analytical method pass into the tubing to the induction furnace from the bubblers. The air stream transports droplets which line the passages and subsequently trap elemental mercury vapor. This mercury source apparently causes intermittent, inexplicable signals in the AAS. The additional sodium borohydride bubbler scrubs off any interferences and droplets, but allows the passage of mercury vapor to the AAS. The second bubbler is used completely empty. It acts as a spray trap and as additional protection for moisture removal from the sample air stream. The basic sample recovery and analytical method utilizing the resistance furnace, modified Hatch and Ott procedure, modified analytical method, induction furnace and AAS was fully successful in processing the silver-treated alumina mercury adsorbent, and the activated charcoal mercury absorbent.

2.8.4 Analysis of Charcoal Absorbent

The activated charcoal used to absorb inorganic and organometallic mercury vapors was analyzed in the same manner as the adsorbent pellets. This method was both practical, from a technical point of view, and economical. It allowed the same equipment developed for the processing and analysis of the pellets to be used for the activated charcoal. The only minor difference in the procedure was that the total charge per canister of charcoal was 75 grams rather than 180 grams. The difference in the weight, of course, is due to the densities and packing characteristics of the two different materials. The charcoal was also processed in 10% aliquots in the resistance furnace, and the collected mercury passed through the GEOMET processing equipment.

2.8.5 Other Analysis Methods

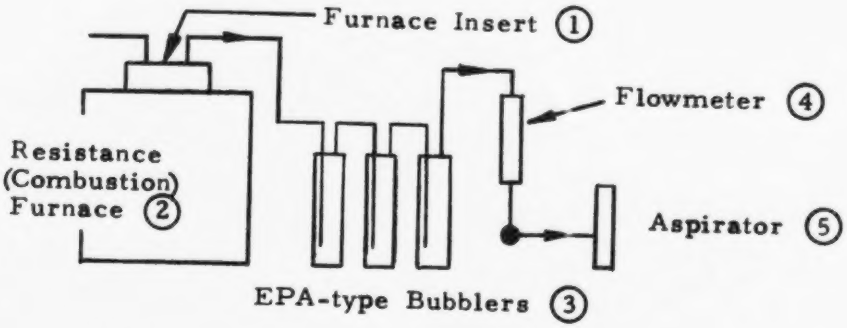
Due to the modification of processing technique for the particulate filters, it seemed appropriate to examine the ICl "soaking" technique for both the silver-treated alumina adsorbent and the activated charcoal absorbent.

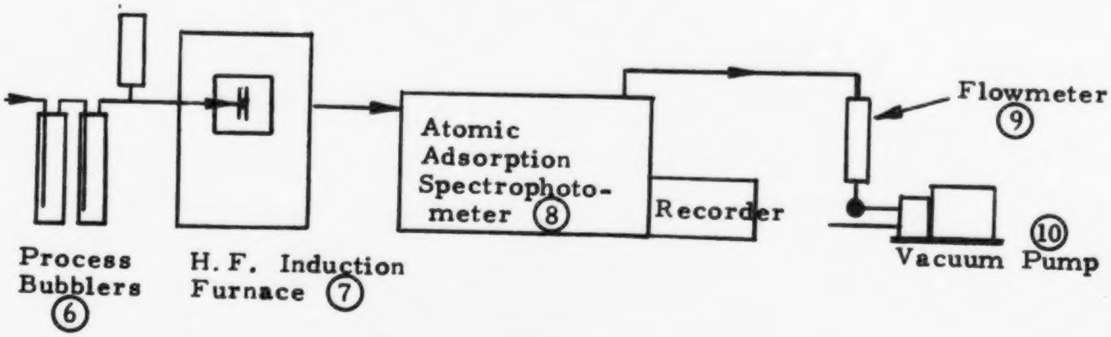
The leeching of mercury from activated charcoal by the ICl solution was not feasible. Bench studies indicated a chemical reaction between the two constituents, and the release of large quantities of iodine. This reaction precludes the use of ICl as a method for chemical removal of the mercury from this absorbent. Other chemical reagents should be evaluated as part of a liquid/solid system for processing of the activated charcoal absorbent by a similar extraction method.

The results from application of ICl to the silver-treated alumina appeared to be more favorable. Trial batches of the alumina were given 30-360 minute soaks in ICl, and this solution was run through the GEOMET processing train. Recovery of elemental mercury appeared low ($\leq$ 50% of collected load) initially, but the method did appear to operate properly with the alumina substrate. Due to the short duration of the program, this potential change was not fully examined. Future evaluation should be carried out with ICl and/or other chemical oxidants/extraction solutions compatible with both the alumina and the activated charcoal absorbent.

2.8.6 Additional Analysis Equipment

The GEOMET processing system for recovery and analysis of all three forms of mercury is fully described in Sections 2.8.2, 2.8.3 and 2.8.4 above. The contract Statement of Work stipulates that other types of equipment suitable for this processing be provided in this report. In order to provide this information with some clarity, the GEOMET processing system can be divided into segments which may then have substitutions of equipment as necessary. The various segments are shown below. Circled numbers in the schematic diagram correspond to entries in the "System Segment and Component" column of the following table:


Furnace Insert ①
Flowmeter ④
Resistance (Combustion) Furnace ②
Aspirator ⑤
EPA-type Bubblers ③


Flowmeter ⑨
Atomic Adsorption Spectrophotometer ⑧
Recorder
⑩ Vacuum Pump
Process Bubblers ⑥
H.F. Induction Furnace ⑦

These segments, and commercially available equivalents, are listed below to aid EPA and the Government in procurement activities.

System Segment and Component	Type	Additional Sources or Substitution Instrumentation
① FURNACE INSERT	GEOMET, Inc.	1) Any qualified machine shop.
② RESISTANCE (COMBUSTION) CRUCIBLE FURNACE	Lindberg, Type 56312-A SOLA Basic Ind. Watertown, Wisc.	1) BLUE-M Electric Co. 138th & Chatham Blue Island, Ill. 60406
③ COLLECTION BUBBLERS	(EPA Design) WEST-GLASS Corp. 12440 Exline St. El Monte, Ca. 91732	1) ACE Glass, Inc. P. O. Box 688 Vineland, N. J.
④ FLOWMETER	Gilmont, Size 12 (0-2 lpm) Roger Gilmont Inst's, Inc. 161 Great Neck Rd. Great Neck, N. Y. 11021	1) FLORATER Flowmeters, Matheson Scientific Co. 1850 Greenleaf Ave. Chicago, Ill. 60007 2) Dwyer VISI-FLOAT Series Dwyer Instruments, Inc. P. O. Box 373 Michigan City, Indiana 46360
⑤ ASPIRATOR, VACUUM PUMP	Nalge #6140 Nalgene Labware Div. Sybron Corp. Rochester, N. Y. 14602	1) AIRJECTOR Fisher Scientific Co. 711 Forbes Ave. Pittsburgh, Pa. 15219 2) CHAPMAN Model, and RICHARD'S Model Aspirators Matheson Scientific Co. (4-1, above)
⑥ PROCESS BUBBLERS	(EPA Design)	1) Reference 3 - 1, above.

Con't.

System Segment and Component	Type	Additional Sources or Substitution Instrumentation
⑦ HIGH FREQUENCY INDUCTION FURNACE	Single Tube, LECO Model 521-000 LECO (Laboratory Equip., Co.) Hilltop Rd. and Leco Ave. St. Joseph, Mich. 49085	1) ECCO High Freq. Co. 7034 Kennedy Blvd. No. Berger, N.J. 07049
⑧ ATOMIC ABSORPTION SPECTROPHOTOMETER	Model 303 PERKIN-ELMER Corp. Main Avenue Norwalk, Conn. 06856	1) Beckman Model 979 Beckman Instruments 2500 Harbor Blvd. Fullerton, Ca. 92634 2) Janell-Ash ATOMSORB; DIAL-ATOM 11; and Model 800 Fisher Scientific (5 - 1, above) 3) IL Model 353 Instrumentation Laboratory, Inc. 113 Hartwell Ave. Laxington, Mass. 02173 4) Corning Model 240 Corning Glass Works Corning, N.Y. 14830
⑨ FLOWMETER	Gilmont, Size 13 (0-12 lpm)	1) Ref. 4 - 1, 2 above.
⑩ VACUUM PUMP	Neptune DYNA-PUMP (0-14 lpm) Universal Elect. Co. Owosso, Mich.	1) Cole-Parmer Inst. Co. 7425 No. Oak Park Ave. Chicago, Ill. 60648 2) Thomas Industries 1419 Illinois Ave. Sheboygan, Wis. 53081

Con't.

SPECIAL NOTES:

Substitution could be made for items ⑦, ⑧, ⑨ and ⑩ by commercially available Mercury Analyzers.

1) GEOMET Model 103
GEOMET, Incorporated
2814-A Metropolitan Pl.
Pomona, Ca. 91767

2) OLIN Mercury Monitor - Liquids or Gas Module
OLIN Custom Analytical Inst's.
120 Long Ridge Rd.
Stamford, Conn. 06904

Substitution could be made for the AAS system, Item ⑧ with commercially available photometer systems.

1) Coleman Mercury Analyzer Model MAS-50
Coleman Inst's. Div.,
PERKIN-ELMER Corp.
42 Madison St.
Maywood, Ill. 60153

2) Mercury Monitor
Laboratory Data Control Co.
Interstate Industrial Park
Riviera Beach, Fla. 33404

3) Model 2006IL Mercometer
Anti-Pollution Technology Corp
937 So. Washington Ave.
Holland, Mich. 49423

2.9 PROTOTYPE SYSTEM TEST DATA

The development methods utilized during the program to obtain a uniform test procedure were oriented to establish a system which would collect the three forms of mercury efficiently for a minimum of twenty-four (24) hours. In addition, bench testing of sample aliquots of absorbents, as well as challenge sources, was used often and with great success. The purpose of this section is to present data obtained on tests which were $\geq$24 hours in duration.

The full Prototype system was challenged with all forms of mercury from standardized sources (Ref. Section 2.4, above). All combinations of challenges were used, i.e. elemental, elemental and organic, the latter combination plus particulate mercury, and finally an ambient air test for Hg in the atmosphere. The tests with laboratory controlled challenges utilized all sources of mercury at levels from 4.5×10^{-2} to 118 $\mu g/M^3$. The ambient air run analysis showed a total collected level of 4.1×10^{-2} $\mu g/M^3$ of elemental mercury averaged over the 25+ hour period. One hour data during this test varied from 12 to 145 nanograms per M^3.

2.9.1 Collection Efficiency Tests

As previously stated, all trial absorbents were bench tested, and monitored by the GEOMET Model 103, to ascertain break-through limitations at various bed depths with variable challenge levels of mercury. This method of testing was used to screen candidate absorbents. Preparational methods and individual sample batches, using the formulation selected as superior (12% Ag/Al_2O_3), were also checked.

Four (4) of the first five of the twenty-four tests were monitored by the Model 103 at the canister exhaust to check for mercury breakthrough. The brief table below indicated the results of these tests.

Run No.	Test Time, Min.	Elem. Hg. Conc.	Collection Efficiency
R2-07	1440 (24 hrs.)	3.8 $\mu g/M^3$	100%
R3-08	1599	28 $\mu g/M^3$	97.1%
R4-09	1440	9 $\mu g/M^3$	99.96%
R5-10	1440	46 $\mu g/M^3$	99.79%
		Average	99.2%

At the end of the period in which tese data were obtained, the silver-treated ($\sim$12%) alumina pellet preparation had been evaluated and adsorbent performance was reproducible. Therefore, it was concluded that the adsorbent pellet collection efficiency was never less than 99.2% and approached 100%. All future testing was done with the Model 103 utilized for monitoring the Hi-Vol exhaust (downstream of the canisters and Hi-Vol blower motor) in order to quantitate the actual challenge level.

Identical procedures were used in establishing the collection efficiency for dimethyl mercury of the Barnebey-Cheney TCA activated charcoal. After a series of bench tests in which dimethyl mercury was undetectable in the downstream gas exhausted from small charcoal canisters by use of the Model 103 in conjunction with a Converter Module, no further measurements were made. Thereafter the collection efficiency was assumed to approach 100%.

The following table is representative of all the 24-hour test runs carried out on the program. It includes the contaminant challenge tests (Runs R19-26 through R22-29), and the ambient air test, Run R23-30. Following the table, Section 2.9.2 presents the analysis of the ambient air test in detail. A discussion of the scatter found in the analysis of pellet samples is included there.

On the basis of the analysis of the entire ambient air test held in 10 batches it was shown that a standard deviation of ±25% is to be expected for an average of ten results. Thus, the material balances obtained for the single results tabulated in Table 2-2 seem reasonable with one or two exceptions.

It is apparent that the major problem involved in achieving reproducible material balances which consistantly approach 100%, relates to sampling the absorbents. When the entire absorbent bed was tested, as in the case of the ambient air test, excellent recoveries were obtainable. Use of 10% aliquots of the solid adsorbent requires very uniform homogenization of the sample. Since about 90% of the mercury is contained on 10-20% of the total sample, it is particularly difficult to obtain representative samples especially when dealing with 1/8" pellets. This problem is resolvable by using the entire solid sample or a larger fraction such as 50%. Minimization of the adsorbent bed volume would assist in facilitating this solution. However the latter adjustment must be compatible with the range of space velocities (volumes of gas/volume of adsorbent per hour) which are acceptable. In general space velocities of the order of 50,000 (vol/vol) per hour have been employed. The upper limit of the range over which this may be acceptably varied is unknown. However, lower values may be employed provided that an absolute quantity of mercury sufficient to satisfy the threshold sensitivity of the analytical method is collected. Further experimentation is recommended.

Table 2-2

PROTOTYPE SYSTEM TEST DATA

	Run Number			
	R2-07	R3-08	R4-09	R5-10
Hi-Vol Air Sampling Rate	40 CFM (1.13 M^3/Min)	44 CFM (1.245 M^3/Min)	40 CFM (1.13 M^3/Min)	40 CFM (1.13 M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)	-----	-----	-----	-----
Elemental Hg Vapor Concentration	3.8 $\mu g/M^3$	28 $\mu g/M^3$	9 $\mu g/M^3$	46 $\mu g/M^3$
Organic Mercury Concentration (Expressed as Elemental Hg)	-----	-----	-----	-----
Sampling Time	1,440 Min.	1,590 Min.	1,440 Min.	1,440 Min.
Canister Sampling Rate	3.56 CFM (3.9%)	4.78 CFM 10.9%)	4.93 CFM (12.2%)	4.73 CFM (11.8%)
Vapor Analysis Rate	1.0 lpm	1.0 lpm	1.0 lpm	1.0 lpm
Analytical Interval	3.9 Min.	3.9 Min.	3.9 Min.	3.9 Min.
Material Balance	62.5%	100.5%	90%	81%

PROTOTYPE SYSTEM TEST DATA (Con't.)

	Run Number			
	R6-11	R7-12	R8-13	R9-14
Hi-Vol Air Sampling Rate	25 CFM (0.71M^3/Min)	23 CFM (0.65M^3/Min)	23 CFM (0.65M^3/Min)	23 CFM (0.65M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)	TEST ONLY, preliminary tests with combination of elemental and organic mercury.		TEST ONLY Organic Mercury	TEST ONLY High Level Elemental
Elemental Hg Vapor Concentration				
Organic Mercury Concentration (Expressed as Elemental Hg)				
Sampling Time	1,165 Min.	1,460 Min.	960 Min.	960 Min.
Canister Sampling Rate	4.85 CFM (19.4%)	4.55 CFM (19.8%)	5.1 CFM (22.2%)	5.2 CFM (22.6%)
Vapor Analysis Rate	1.0 lpm	1.0 lpm	1.0 lpm	1.0 lpm
Analytical Interval	3.9 Min.	3.9 Min.	3.0 Min.	3.9 Min.
Material Balance	N/A	N/A	N/A	N/A

PROTOTYPE SYSTEM TEST DATA (Con't.)

	Run Number			
	R10-15	R11-16	R12-17	R13-18
Hi-Vol Air Sampling Rate	LAB TESTS ONLY Improved Recovery Procedures.		20 CFM (0.57M^3/Min)	15 CFM (0.42M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)			0.33 μg	4.25 mg
Elemental Hg Vapor Concentration			0.4 μg/M^3	1.92 μg/M^3
Organic Mercury Concentration (Expressed as Elemental Hg)			118 μg/M^3	0.6 μg/M^3
Sampling Time			1,440 Min.	1,440 Min.
Canister Sampling Rate			4.65 CFM (23.8%)	3.46 CFM (23.2%)
Vapor Analysis Rate			4.0 lpm	4.0 lpm
Analytical Interval			3.0 Min.	3.0 Min.
Material Balance			86% Avg.	>98.5% Avg.

PROTOTYPE SYSTEM TEST DATA (Con't.)

	Run Number			
	R14-19	R15-20	RS13X-21	R16-22
Hi-Vol Sampling Rate	22 CFM (0.62M^3/Min)	22 CFM (0.62M^3/Min)	19 CFM (0.54M^3/Min)	22.5 CFM (0.64M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)	92.2 mg*	8.6 mg*	--0--	--0--
Elemental Hg Vapor Concentration	69.1 $\mu g/M^3$	53.3 $\mu g/M^3$	4.4 $\mu g/M^3$**	4.5x10^{-2} $\mu g/M^3$*
Organic Mercury Concentration (Expressed as Elemental Hg)	16.0 $\mu g/M^3$	7.3 $\mu g/M^3$	2.6 $\mu g/M^3$	6.7x10^{-4} $\mu g/M^3$*
Sampling Time	1,440 Min.	1,410 Min.	1,020 Min.	1,380 Min.
Canister Sampling Rate	4.80 CFM (21.9%)	5.36 CFM (24.4%)	3.62 CFM (19.0%)	4.88 CFM (21.7%)
Vapor Analysis Rate	0.16 lpm	0.17 lpm	4.0 lpm	36 lpm
Analytical Interval	3.6 Min.	3.6 Min.	3.0 Min.	6.0 Min.
Material Balance	148%	91.5%	57.5%**	Sample Contaminated.

* Calculated

** Molecular Sieve Run

PROTOTYPE SYSTEM TEST DATA (Con't)

	Run Number			
	R17-23	R18-24	RS13X-25	R19-26 (I)
Hi-Vol Air Sampling Rate	21.5 CFM (0.61 M^3/Min)	20 CFM (0.57 M^3/Min)	19 CFM (0.54 M^3/Min)	21 CFM (0.59 M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)	---0---	4.31 mg*	14.23 mg*	---0---
Elemental Hg Vapor Concentration	2.0 $\mu g/M^3$	78 $\mu g/M^3$	90 $\mu g/M^3$	7.9 $\mu g/M^3$
Organic Mercury Concentration (Expressed as Elemental Hg)	0.57 $\mu g/M^3$	6.7 $\mu g/M^3$	12.2 $\mu g/M^3$	2.6 $\mu g/M^3$
Sampling Time	1,548 Min.	1,350 Min.	1,470 Min.	435 Min.
Canister Sampling Rate	5.22 CFM (24.3%)	4.95 CFM (24.7%)	3.68 CFM (19.4%)	4.82 CFM (23.0%)
Vapor Analysis Rate	36 lpm	2 lpm	1.9 lpm	4 lpm
Analytical Interval	6.0 Min.	3.6 Min.	3.6 Min.	3.0 Min.
Material Balance	126%	90%	46.5%**	99.9%
Added Contaminants/Concentration	----- -----	----- -----	----- -----	Chlorophenol (0.5 ppm) Xylene (19.5 ppm)

*Calculated; **Molecular Sieve Run; (I) Interference Test

PROTOTYPE SYSTEM TEST DATA (Con't.)

Run Number

	R20-27 (I)	R21-28 (I)	R22-29 (I)	R23-30 (A)
Hi-Vol Air Sampling Rate	22 CFM (0.56 M^3/Min)	20.5 CFM (0.56 M^3/Min)	22 CFM (0.56 M^3/Min)	23 CFM (0.65 M^3/Min)
Particulate Mercury Concentration (Expressed as Elemental Hg)	---0---	---0---	---0---	---0---
Elemental Hg Vapor Concentration	6.1 $\mu g/M^3$	8.4 $\mu g/M^3$	8.0 $\mu g/M^3$	4.1 x 10^{-2} $\mu g/M^3$
Organic Mercury Concentration (Expressed as Elemental Hg)	0.55 $\mu g/M^3$	0.67 $\mu g/M^3$	19 $\mu g/M^3$	---0---
Sampling Time	1,015 Min.	1,015 Min.	947 Min.	1,540 Min.
Canister Sampling Rate	5.02 CFM (24.0%)	5.02 CFM (24.0%)	4.86 CFM (22.1%)	4.74 CFM (20.6%)
Vapor Analysis Rate	2 lpm	2 lpm	2 lpm	82 lpm
Analytical Interval	3.0 Min.	3.0 Min.	3.0 Min.	6.0 Min.
Material Balance	87.5%	70.0%	53.5%	98.8%
Added Contaminants/Concentration	NO gas (1.6 ppm)	SO_2 gas (1.7 ppm)	H_2S gas (2.5 ppm)	----- -----

(I) Interference Test

(A) Ambient Air Test

2.9.2 Ambient Air Monitoring for Elemental Mercury

As a check of the performance of the method of mercury collection and analysis utilizing the canisters and accessories as developed during the contract, an ambient air test for elemental mercury was carried out from the roof of the Pomona Laboratory.

The apparatus was set up as previously described (Section 2.5 and 2.7). The air sample from out-of-doors was ducted through a three inch (i.d.) flexible stainless sttel tube to the manifold at the top of the Hi-Vol Sampler. The GEOMET Model 103 was connected, as previously, to the exhaust from the plenum section of the Hi-Vol assembly.

The ambient air test was carried out for 25 hours and 40 minutes (1540 minutes) with 180g of Ag/Al_2O_3 pellets in the canister used for elemental mercury collection. The pellets utilized were those which had proved superior in previous testing, Harshaw alumina Grade Al-0104T impregnated with 12% silver. The preparational method has been described in Section 2.6.3. The GEOMET Model 103 was operated on a 6-minute cycle with 257 analyses automatically obtained and printed during the course of the experiment. The following table shows the conditions and the Model 103 results.

Conditions

Test Duration	1540 min.
Hi-Vol Sampling Rate	651 lpm
Canister Sampling Rate	134 lpm (20.6% of total)
Model 103 Sampling Rate	82 lpm

Test Results from GEOMET Model 103

Number of Analyses	257
High Hourly Value (10 analyses/hr)	145 ng/m^3-hr.
Low Hourly Value (10 analyses/hr)	12 ng/m^3-hr.
Average Value (25+ hours)	41 ng/m^3

Tables 2-3 and 2-4 show the individual results obtained with the Model 103; Figure 2-42 shows the applicable calibration.

After mixing by rolling and quartering methods, the 180 g canister sample of pellets was separated into ten batches of 18 g of pellets each. These were analyzed by the techniques indicated in Section 2.8.3. In general, a 5.0 ml aliquot of the 45 ml iodine monochloride absorbent was used in the reduction step. The results were obtained by use of a Perkin-Elmer Model 303 atomic adsorption spectrophotometer. Calibration of this instrument is shown in Figure 2-43.

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```
0000044 — 50        Time 2015 Hours, 2/26/73
0000045
0000046
0000048
0000047
0000052
0000055
0000054
0000048
0000056
0000056 —— 79
0000051
0000053
0000059
0000059
0000074
0000086
0000111
0000111
0000132 —— 215
0000135
0000163
0000189
0000189
0000224
0000231
0000231
0000255
0000252
0000282 —— 298
0000282
0000284
0000284
0000311
0000303
0000304
0000311
0000335
0000378
0000185             Time 1615 Hours, 2/26/73
```

Numbers in right hand margin are averages of DVM readings for ten previous printouts.

Table 2-3

Analysis on Ambient Air Test
GEOMET Model 103
Air Sampling Rate 82 1/m

Read Results Upward from Bottom to Top

Time 0051 Hours, 2/27/73

```
0000039
0000039
0000037
0000034
0000032
0000036  38
0000037
0000038
0000037
0000038
0000037
0000039
0000036
0000039
0000037
0000040  37
0000040
0000039
0000027
0000039
0000041
0000041
0000035
0000040
0000040
0000031  35
0000031
0000040
0000040
0000032
0000032
0000036
0000042
0000043
0000043
0000048  43
0000047
0000042
0000042
0000046
0000046
0000049
0000046
0000033
0000033
0000042
```

Time 2015 Hours, 2/26/73

Time 0509 Hours, 2/27/73

```
0000033
0000030
0000030
0000034
0000034
0000037
0000035
0000035
0000035   34
0000032
0000034
0000034
0000033
0000035
0000034
0000036
0000036
0000029
0000033   37
0000042
0000039
0000033
0000035
0000040
0000034
0000034
0000035
0000040
0000039   35
0000038
0000025
0000025
0000035
0000032
0000039
0000035
0000040
0000040
0000038
0000032   35
0000032
0000036
0000036
```

Time 0051 Hours, 2/27/73

Time 0945 Hours, 2/27/73

```
0000035
0000032
0000029
0000029
0000025
0000027   31
0000033
0000037
0000036
0000035
0000035
0000028
0000027
0000026
0000026
0000030   27
0000022
0000021
0000030
0000028
0000028
0000025
0000029
0000027
0000032   33
0000032
0000031
0000031
0000033
0000035
0000035
0000036
0000035
0000035
0000031
0000037   32
0000033
0000030
0000030
0000031
0000032
0000026
0000026
0000034
0000036
0000033   34
```

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Time 0509 Hours, 2/27/73

Time 1357 Hours 2/27/73

```
0000209
0000221
0000223
0000223
0000211
0000194
0000199 — 214
0000191
0000215
0000219
0000225
0000214
0000222
0000224
0000216
0000213
0000203
0000180 — 154
0000176
0000176
0000164
0000159
0000146
0000146
0000138
0000131
0000125
0000122 — 77
0000122
0000100
0000096
0000076
0000059
0000059
0000046
0000043
0000048
0000038 — 34
0000038
0000039
0000037
0000034
```

Time 0945 Hours, 2/27/73

Time 1755 Hours, 2/27/73

```
0000110
0000110   787/7 = 112
0000105
0000100
0000115
0000118
0000129
0000133 — 148
0000138
0000129
0000137
0000137
0000140
0000151
0000168
0000177
0000173
0000173 — 154
0000171
0000159
0000159
0000128
0000128
0000168
0000166
0000166
0000168
0000175 — 182
0000180
0000181
0000181
0000192
0000189
0000181
0000185
0000185
0000176
0000169 — 206
0000191
0000220
```

Time 1357 Hours, 2/27/73

Table 2-4

Atmospheric Test
GEOMET Model 103 Data*
Started 1615 hrs., 2/26/73

Reading (DVM divs)	Analysis (ng/m^3)	Time (hrs., midpoint)
298	145	1645
215	99	1745
79	33	1845
50	20	1945
43	18	2045
39	16	2145
37	15	2245
38	16	2345
35	15	0045 (2/27)
35	15	0145
37	15	0245
34	15	0345
34	15	0445
32	14	0545
33	14	0645
27	12	0745
31	14	0845
34	15	0945
77	32	1045
154	67	1145

---

*Each reading represents an average of 10 readings.

Table 2-4 (Con't.)

Reading (DVM divs)	Analysis (ng/m^3)	Time (hrs., midpoint)
214	99	1245
206	94	1345
182	82	1445
159	70	1545
148	64	1645
112 (7 readings)	48	1706

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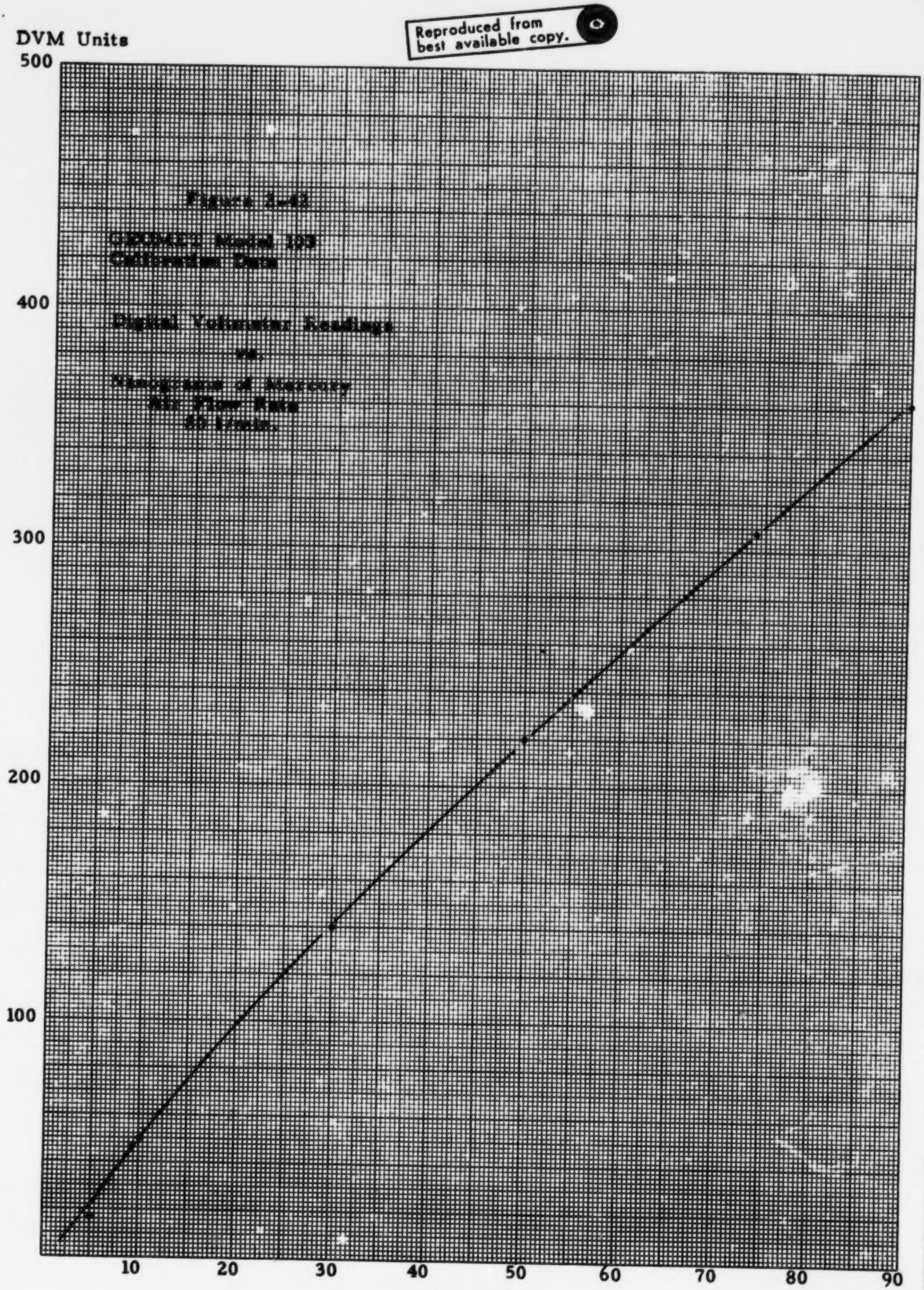

Figure 2-42

GEOMET Model 103
Calibration Data

Digital Voltmeter Readings
vs.
Nanograms of Mercury
Air Flow Rate
40 l/min.

K+E 10 X 10 TO THE CENTIMETER 46 1513
18 X 25 CM MADE IN U.S.A.
KEUFFEL & ESSER CO.

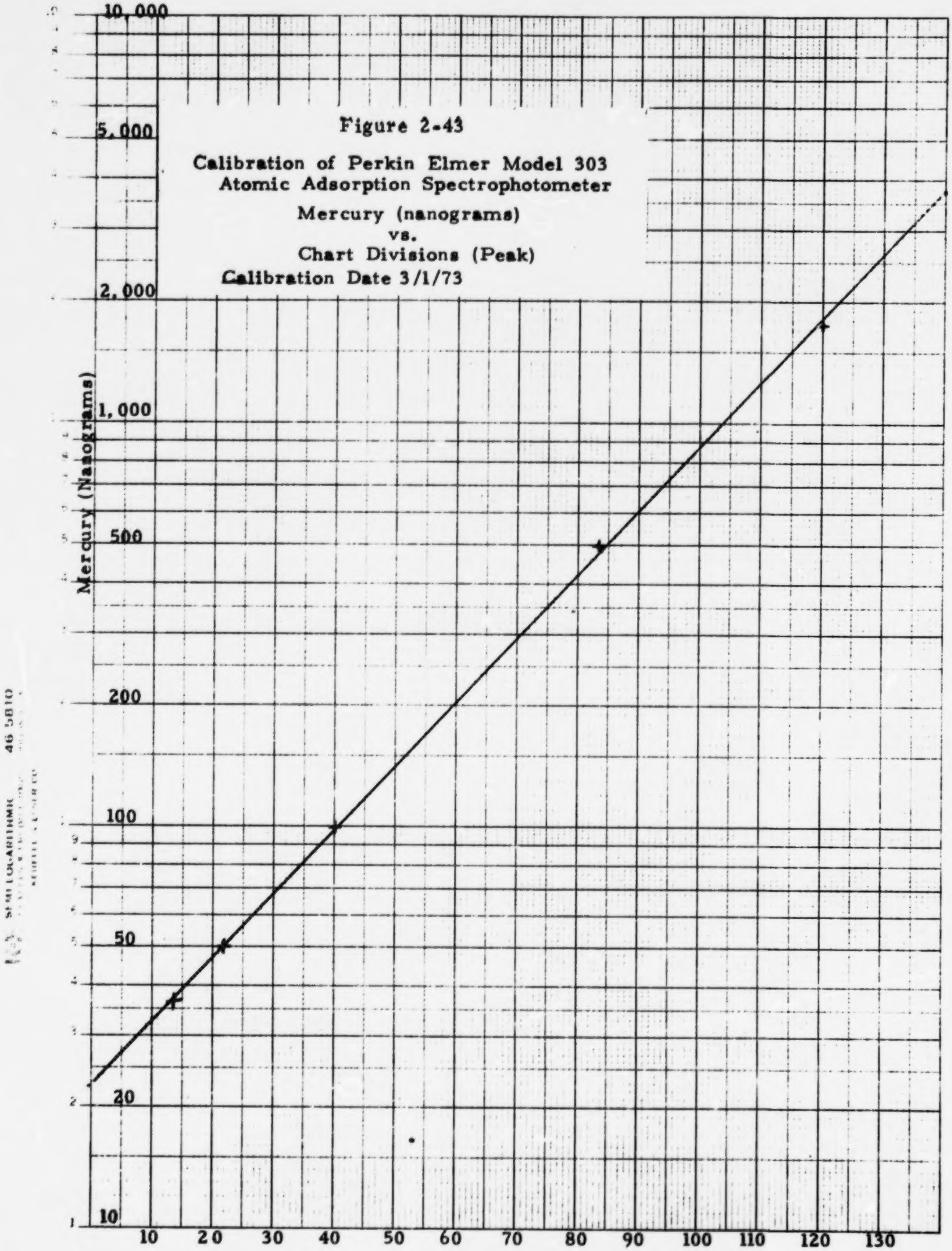

Figure 2-43

Calibration of Perkin Elmer Model 303
Atomic Adsorption Spectrophotometer
Mercury (nanograms)
vs.
Chart Divisions (Peak)
Calibration Date 3/1/73

The results for the ten portions of the adsorbent are shown in Table 2-5. As is apparent from the average air concentrations calculated from both methods and the total amounts of mercury as analyzed by both methods, the High-Vol canister procedure and the GEOMET 103, closely correspond. The canister procedure showed an average air concentration of 40.9 ± 10.5 ng/m^3. A simple standard deviation cannot be calculated for the GEOMET data since the concentration of mercury in the air sample changes with time.

However, the standard deviation associated with the ten determinations of the canister sample indicates a need for improvement. The 25.6% standard deviation indicates that mixing and sampling of the 180 g adsorbent bed has not been adequately achieved. Two alternatives seem appropriate: (1) the adsorbent bed size should be reduced so that (2) a relatively large fraction (or all) of the adsorbent may be used in the recovery analysis. As a third choice a more rigorous procedure for mixing the solid sample may be instituted. However since a very large fraction of the total mercury is retained at the top of the adsorbent, particles from the first layer will always have a disproportionate effect on an analysis. Thus, it would be preferable to analyze the entire adsorbent sample

Table 2-5

Ambient Air Test, Canister Method.

Analysis of Silver/Alumina Adsorbent

Sample No.	Total Hg Collected (ng)		Deviations (ng)
1	1260		415
2	720		125
3	783	756 Average	62
3 Replicate	729		116
4	567	585 Average	278
4 Replicate	603		242
5	666		179
6	810		35
7	1008		163
8	945		100
9	774		71
10	927		82
Total	8451 nanograms		1868
Average	845 ±		186.8 (a. d.)(22%)

Air Sampled	206.4 m^3
Average Air Concentration	40.9 ng/m^3 ± 10.5 (σ) ng/m^3
Standard Deviation of Total	216 (25.6%)
Mean Deviation of Total	187 (22.1%)

Section 3.0

CONCLUSIONS, RECOMMENDATIONS AND COMMERCIAL PRICE ESTIMATES

3.1 SUMMARY

During the course of the program a prototype device for the quantitative collection of airborne mercury in particulate, elemental and combined forms, respectively, was designed, fabricated and tested. The device utilized a Hi-Vol Sampler into which two canisters containing absorbers for elemental and combined mercury were added in a collection plenum below the glass fiber filter. The plenum was located between the funnel-shaped inlet duct of the Hi-Vol Sampler and the blower motor. It supports the two collection canisters and controls the air flow through the series arrangement. Of the total air (20+ CFM), about 1/5 passes through the canisters.

Particulate collection was tested with mercuric oxide and sulfide on the glass fiber filter; elemental mercury vapor was collected on a silver-alumina adsorbent; dimethyl mercury was collected on the activated charcoal phase.

As evaluated, the preferred configuration of the first canister contained 180 g (160 ml) of 12% silver on alumina (Harshaw, Al-0104T) in a 1.56" (i.d.) x 5.5" cyclinder. This adsorber removed 99.9+% of the elemental mercury passed through in air samples. The second canister (same dimensions) utilized 75 g (160 ml) of activated charcoal (Barnebey-Cheney, TCA grade). It removed all combined mercury from the gas streams. Other absorbents and configurations were also examined.

The absorbents were performance tested in 24-hour runs at challenge levels from ambient to 118 $\mu g/m^3$. In addition, tests were carried out in the presence of gaseous pollutants: hydrogen sulfide, sulfur dioxide, chlorophenol, nitric oxide and xylene. None of the pollutants modified the quantitative collection performance of the absorbents.

As designed, the prototype canisters are made of PVC pipe. Each is closed by a stainless screen held in place by a Lucite fitting which is also used to stack the canisters. An aluminum plenum houses the added assembly. The collection canisters may be emptied directly into a Pyrex shipping container or each canister may be enclosed in a sealed polyethylene shipping container and returned to a central laboratory for analysis and refilling. Tests have shown stable retention of mercury and its compounds by the absorbents for periods up to six weeks.

Particulate samples are stored by carefully folding the glass fiber particle filter and enclosing it in a polyethylene container for mailing and storage.

An analytical procedure for each of the three separately collected forms of mercury has been developed and tested. In principle, it involves desorbing the collected sample into iodine monochloride solution by application of heat to the sample; reduction of an aliquot of the ICl solution; collection of the resultant elemental mercury on gold wire; heating the gold wire by an induction furnace to desorb the concentrated mercury into a flameless atomic absorption spectrophotometer cell; and measurement of the desorbed mercury by standard AAS light absorption techniques at 253.7 nm. A number of variations of the method are possible.

Data have been enclosed to support all claims. A prototype device was also developed for the sample transfer operation. The latter consists of a crucible furnace and furnace insert to transfer the mercury-containing gases to ICl bubblers.

Detailed designs (drawings) of all developed parts are included.

On 21 February, a demonstration of the operation of the collection device was conducted before the Project Officer in the Pomona laboratory.

3.2 RECOMMENDATIONS

Recommendations to improve the field utility, convenience and operating costs include the following:

(1) It would be highly desirable to reduce the volume, weight and potential cost of the PVC canisters and the absorbents contained in each. Two approaches to resolution of this need might be conceived: (a) the volume of adsorbents may be reduced by careful determination and specification of the absorption capacity requirement, air sample volume requirements, sampling time and collection efficiency requirements; (b) two canister sizes might be developed for industrial monitoring; a second for ambient determinations. A series of interacting technical factors are involved in size reduction including the efficiency and composition of the adsorbents and the factors indicated above.

Ancillary advantages to decrease in canister size include reduction of the size of the plenum, improved ease in shipping, increased convenience and precision in sample analysis, etc.

(2) Cost reductions may be achieved by development of a less expensive adsorbent than the currently utilized 12% silver on alumina. It is anticipated that reduction in silver content might be achieved by definition of adsorption capacity, efficiency factors and operating time so that a lesser safety margin than utilized in the current adsorbent is specified. Also, new adsorbents may also be employed.

(3) Operational expense may be considerably decreased by development of methods for quantitatively regenerating the adsorbents.

(4) An area where considerable simplification in the analytical method might be gained is related to recovery and extraction of mercury - containing materials and transfer into iodine monochloride solutions. Currently this transfer is achieved by heating samples of absorbents in a furnace while simultaneously drawing air through the furnace into ICl bubblers. This procedure works well enough, but direct extraction of each sample into liquid offers considerable simplification. Initial tests with the particulate filter and silver on alumina adsorbent indicate that both these two samples appear tractable by liquid extraction. Use of sonicators or automatic shakers appears useful in assisting in achievement of satisfactory recovery results.

This procedure would reduce the amount of labor involved in each analysis, eliminate problems associated with manipulations in a heated furnace, and potentially increase the precision of the sample transfer process.

3.3 COMMERCIAL PRICE ESTIMATES

For purposes of reference only, the following estimates of commercial sale prices for the components and services developed under this project have been estimated. These price estimates include standard commercial profit margins and are subject to change for volume sales and, of course, in the event of technical improvements and simplifications.

Material	Estimated Market Price
(a) Collection Assembly: Designed for use with standard Hi-Vol Air Samplers. Includes stainless steel plenum, canister holder, critical orifice and air flow controls but without canisters or pressure gage; complete with directions for use.	$145.00
(b) Elemental Mercury Collection Canisters: Filled with 180 grams of 10% silver on 1/8" alumina adsorbent pellets.	$ 49.50
(c) Combined Mercury Collection Canisters: Filled with 85 grams of 6-10 mesh activated charcoal.	$ 22.50
(d) Particulate Collection Filters: Glass Fiber Filters, 8 x 10 inches, for use with standard Hi-Vol samplers. In packages of 100 sheets.	$ 21.50

Material	Estimated Market Price
(e) Shipping Containers: Wide mouth Nalgene bottles with screw caps, 48 mm opening. For shipment of canisters or folded filters after presealing in plastic envelopes (not included).	$ 7.50 per dozen
Analyses:	
(a) Certified analysis, refilling and return of Elemental Mercury Canisters	$ 35.00
(b) Certified analysis, refilling and return of Combined Mercury Canisters.	$ 30.00
(c) Certified analysis of Particulate Samples on 8 x 10 inch glass fiber filters.	$ 22.50

These estimates are presented for reference and should be utilized as the bases for measurement of improvements.

Section 4.0

EQUIPMENT DEVELOPMENT TABULATION

The equipment developed under this contract consists of elements of the test system, preliminary and Prototype collection plenums, Prototype canisters and segments of the GEOMET mercury recovery and analysis.

The following tabulation, Table 4-1, lists these items along with their type, source and referenced drawings contained within the text of this report.

Table 4-1

EQUIPMENT TABULATION SHEET

Item	Type	Source	Reference Section
1. High-Volume Air Sampler with Shelter (1 each).	Unico 550 Turbine Jet; with Wooden Shelter.	Environmental Science Div., Bendix Corp.	2.3
2. Collection Plenum, Test. (1 each)	GEOMET; with Probe for Mercury Challenge Monitoring.	GEOMET, Inc.	2.5, 2.7.2
3. Collection Plenum, Prototype configuration (1 each).	GEOMET; with Critical Orifice for Monitoring Canister Through-put Air Velocity.	GEOMET, Inc.	2.7.2
4. Dilution Air System (1 each).	GEOMET; Includes Flowmeters, Air Filter Canister, MAGNEHELIC Gage, and Air Pump.	GEOMET, Inc.; Dwyer Instruments Inc.; Thomas Industries, Inc.	2.5
5. Prototype Absorbent Canisters (6 each).	GEOMET; PVC plastic.	GEOMET, Inc.	2.7.3
6. Prototype Canister Closures (12 each)	GEOMET; LUCITE plastic and Stainless Steel Screen.	GEOMET, Inc.	2.7.3

Table 4-1 (Con't.)

EQUIPMENT TABULATION SHEET

Item	Type	Source	Reference Section
7. Crucible Furnace (1 each).	Lindberg HEVI-DUTY, Type 56312-A.	Lindberg Division, (SOLA Industries)	2.7.4
8. Furnace Insert (1 each)	GEOMET; with two (2) interchangeable Nickel Alloy Crucibles.	GEOMET; Van Waters and Rogers Co.	2.7.4